UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION II

DATE: MAR 2 9 2006

SUBJECT: Review of the Draft Remedial Investigation / Feasibility Study Work Plan, Operable Unit 3, for the Cornell-Dubilier Electronics Superfund Site, South Plainfield, New Jersey

FROM: Sergio López-Luna, P.E.
Hazardous Waste Support Section (2DESA-HWSB)

TO: Peter Mannino, Remedial Project Manager
Central New Jersey Remediation Section (2ERRD-NJRB)

At your request, the Hazardous Waste Support Section (HWSS) has reviewed the Operable Unit 3 (OU3) Draft Remedial Investigation / Feasibility Study (RI/FS) Work Plan (WP), dated February 2006, and received by the HWSS February 16, 2005, for the Cornell-Dubilier Electronics Superfund Site, South Plainfield, NJ. This document also contained the Field Sampling Plan (FSP) / Quality Assurance Project Plan (QAPP) as Attachment A, a Health and Safety Plan (HASP) as Attachment B, and the Preliminary Conceptual Site Model (CSM) Report (Revised) as Attachment C. These documents were prepared for the Dana Corporation by HydroQual Inc., Mahwah, NJ, Environ Corporation, Princeton, NJ, and de maximis, inc., Clinton, NJ. Our comments for the WP and FSP / QAPP, less the HASP and CSM, are attached.

We would appreciate receiving a copy of your correspondence to the respective parties transmitting EPA comments on these documents. If you have any questions or require further information, please contact me at (732)321-6778 or James Marshall-Zank at (732)321-4438.

Attachments

cc: John Prince, 2ERRD-NJRB

298887

Draft RI/FS Work Plan - Operable Unit 3 for the
Cornell-Dubilier Electronics Superfund Site, South Plainfield, New Jersey

General Comments on the Draft Work Plan

1. It is stated in Section **2.6 PUBLIC/RESIDENTIAL WELL SEARCH**, page 2-7, as part of the ongoing evaluation of well pumping data, that HydroQual, Inc. sent a letter of request for assistance in reconciling inconsistencies and filling information gaps, to the Middlesex Water Co. in December, 2005, with no response. It should be clarified if it is known whether the Middlesex Water Co. received the letter, and the significance of the RI/FS missing data to the project.

2. It is stated in Section **4.2.2.1 Overview** that the fieldwork conducted for this investigation will be performed using the Triad Approach, which calls for a dynamic work plan that is based upon established SOPs and decision criteria for the collection and evaluation of field screening data. It should be clarified that the Triad involves three main components: (1) systematic project planning, (2) dynamic work strategies, and (3) real-time measurement technologies. Although many elements of these features are located within the Work Plan, it is important to note that the QA Officer needs to be aware of real-time measurement technology-specific QC requirements, and also that data quality must be assessed and tracked as data are generated.

3. In Section **4.2.2.3 Groundwater Investigation**, page 4-6, it is stated that total volatile organic concentration (TVO) profiling will be accomplished by field screening of groundwater samples using the Color-Tec method marketed by A.P. Buck, Inc. It should be noted how some of the disadvantages of this method will be addressed. In addition, it should be noted that this method is not located on the USEPA Environmental Technology Verification (ETV) Program listing of verified technologies, or at the Superfund Innovative Technology Evaluation (SITE). The ETV listing is available at http://www.epa.gov/etv, and the SITE monitoring and measurement technologies at http://www.epa.gov/ORD/SITE.

4. For Section **4.4 TREATABILITY STUDIES (SOW TASK V)**, pages 4-26 through 4-29, a useful reference is the document, *EPA Guidance for Conducting Treatability Studies under CERCLA, EPA/540/R-92/071a, October 1992*, located at http://www.epa.gov/superfund/resources/remedy/pdf/540r-92071a-s.pdf.

Specific Comments on Attachment A: Field Sampling Plan (FSP) / Quality Assurance Project Plan (QAPP)

1. It should be noted that the US DOD, EPA, and DOE implemented the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP), March 2005, as the standard format for preparing QAPPs for Federal Facilities. USEPA Region 2 has adopted this guidance for all Superfund and RCRA projects within the Region in November 2005. Any QAPPs previously prepared do not have to follow the UFP format, however, all new and significantly revised generic and site-specific QAPPs prepared for sites in Region 2 will need to follow this new format. Approved QAPPs require an annual review and updating as necessary. QAPPs over five years old require revision and resubmittal. The various UFP-QAPP documents are located at http://epa.gov/fedfac/documents/qualityassurance.htm.

2. It should be noted that USEPA Region 2 requires the *Analytical Services Tracking System* (ANSETS) reporting requirements which utilize the "ANSETS Data Requirement" form for Non-CLP analytical data effective January 1, 2003 instead of the "Non-CLP Tracking Form" previously used. The basis for this is in the USEPA OWSER Memorandum, *Tracking Superfund Non-CLP Analytical Data, Directive # 9240.0-2C, November 14, 2002* and the accompanying *USEPA Region 2 Memorandum, same title, issued in January, 2003.* A copy of the Region 2 memorandum is attached. Per the OWSER memorandum, detailed instructions and procedures for submitting ANSETS data can be found by contacting the USEPA Region 2 Regional Sample Control Coordinator (RSCC), Ms. Jennifer Feranda at (732) 321-6687 or feranda.jennifer@epa.gov, or Mr. Adly Michael at (732) 906-6161 or michael.adly@epa.gov. In addition, further information is located at http://www.epa.gov/superfund/programs/clp/ansets.htm.

3. **A4 Project / Task Organization and Schedule.**

a. **USEPA**, page 8 of 14. It is stated in the second paragraph that the Region 2 QA Reviewer, to be designated by USEPA, will be responsible for the review and approval of the FSP/ QAPP associated with this project. It should be clarified that the Region 2 Hazardous Waste Support Section (HWSS) QA Reviewer will submit comments to the Remedial Project Manager (RPM), who will determine the final approval.

b. **HydroQual, Inc.**, page 9 of 14. It is stated that Mr. Timothy R. Roeper, P.G., will serve as the Project Manager for the RI and in that role will also function as the QA Officer. Mr. Roeper will manage implementation of the work plan activities for the OU-3 investigation. This is also demonstrated in Figure A4-1, Project Organization Chart. Per the documents *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, March 2001* located at http://www.epa.gov/quality, and *IDQTF UFP-QAPP Manual, March 2005* located at http://epa.gov/fedfac/documents/qualityassurance.htm, it should be verified that the QA Officer (Manager) is independent of those generating the data and collection activities and works separately from those performing project tasks. In addition, generally the QA Manager is the person who is responsible for maintaining the official, approved QAPP. This should be further clarified and corrected.

4. **A7.1 Project Quality Objectives**, page 12 of 14.

a. It should be noted that the USEPA's 7-step Data Quality Objective (DQO) process and activities is used during the remedial investigation of hazardous waste sites and this is the recommended systematic planning approach for data collection activities. This will provide a sampling design that will accomplish the goals of this project and support decision-making. EPA guidance on the DQO process is contained in *EPA QA/G-4HW, Data Quality Objectives Process for Hazardous Waste Site Investigations, January 2000*, and *EPA QA/ G-4 Guidance for the DQO Process, August 2000*, both at http://www.epa.gov/quality, and additional information is located at http://www.hanford.gov/dqo/. Under the UFP-QAPP format, it is stated that when critical environmental decisions need to be made (e.g., final decision-making or compliance with a standard), a formal systematic planning process such as the DQO process should be followed.

As an example, if the objective is to assess current groundwater quality conditions, and whether the impact of the groundwater quality poses a potential threat to human health, welfare, or the environment, and to measure the overall effectiveness of the remedial action, then the decision-making process should be used to determine if (1) further groundwater cleanup is necessary, (2) additional investigation is needed before a decision can be made, or (3) no further action is required. This should be reviewed so that appropriate corrections can be made.

b. It is stated at the end of the third paragraph that a subset of samples may be analyzed for a list of several parameters including ammonium. In addition, ammonium appears in Tables A7-1 and A7-3, both on page 5 of 5, the last page of Table A7-2, with the cited EPA Methods 350.1 and 350.2, and in Tables located at the end of Section B. It should be clarified that the referenced methods specify ammonia-nitrogen, and not ammonium. It should be noted that ammonium occurs as a cation in solution (ammonium ion, NH_4^+), or as an ammonium salt such as ammonium chloride, etc. This should be corrected.

5. **A8 Special Training Requirements and Certification**, page 14 of 14. It is stated that at least one individual will be trained in the operation and recording of data obtained from the Color-Tec field screening method. In addition, for field analysis of soil gas samples, it is stated that at least one individual will be trained in the calibration and operation of the field GC/MS. It should be noted given the importance of the expected data from the Color-Tec field screening method, along with the field analysis and the complexity of a GC/MS, that more than one individual should be trained for each procedure given the field dynamics of the Triad approach. For example, it is documented in literature that operator error potential exists for the Color-Tec method due to airborne contaminants, subtle color change at low concentrations, and false positives or negatives from moisture and interference compounds that could impact results. If the only trained operator becomes unavailable, the sampling would be interrupted since an untrained or inexperienced operator could not be used. This should be addressed.

6. **B2.3 Groundwater Sample Collection**, page 9 of 39. It is stated in the fourth paragraph that groundwater samples collected from typical two-inch diameter or larger monitoring wells will be done in accordance with the Low Flow / Purge sampling methodology as described in the Groundwater Sampling SOP in Appendix A. In referencing this SOP, page 1, first paragraph, it is stated that monitoring wells will be purged in accordance with the USEPA Region 2 SOP "Groundwater Sampling Procedure Low Stress (Low Flow) Purging and Sampling" (March 16, 1998), included as an attachment to this SOP. It should be noted, however, that the cited EPA Region 2 SOP could not be located as an attachment. It is available at http://www.epa.gov/Region2/desa/hsw/lowflow.pdf. This should be corrected.

7. **B2.4 Cleaning and Decontamination**, pages 10-11 of 39.

a. It is stated that cleaning and decontamination of equipment used for sample collection will be conducted in accordance with the Equipment Decontamination SOP (Appendix A). It should be noted that the decontamination procedures within the cited USEPA Region 2 SOP, "Groundwater Sampling Procedure Low Stress (Low Flow) Purging and Sampling" (March 16, 1998), comment No. 6, above, vary from those contained in the Equipment Decontamination SOP (Appendix A). This should be reexamined.

b. In addition to the cited Method TO-15, a Method TO-15 Supplement - Analysis of 1,1-DCE at pptv Concentrations is available, and attached.

c. It should be stated what will be the disposition of any Investigation Derived Wastes (IDW). The documents *Guide to Management of Investigation-Derived Wastes, Pub. 9345.3-03FS, January 1992*, located at http://www.epa.gov/superfund/resources/remedy/pdf/93-45303fs-s.pdf and *Guide to Discharging CERCLA Aqueous Wastes to Publicly Owned Treatment Works (POTWs)*, located at http://www.epa.gov/superfund/resources/remedy/pdf/93-30213fs-s.pdf may be of assistance.

8. **B3.1 Requirements and Provisions for Sample Handling.**

a. Sample Containers and Preservatives, page 12 of 39. It should be noted that a reference for the selection of sample containers is the document *Specification and Guidance for Obtaining Contaminant-Free Sample Containers, EPA 540/R-93/051 and OSWER Directive 9240.0-05A(EPA,1992b)*. If this document cannot be located, please contact the USEPA RPM.

b. Sample Shipment, page 16 of 39. In addition to the information provided in the last paragraph of this subsection, it should be noted that HMR; 49 CFR Parts 171-180, may also apply. The attached letter from the USDOT pertaining to this subject may be helpful when shipping environmental samples.

9. **B5.2 Laboratory Quality Control**, Statistical Determination of Precision and Accuracy, pages 24-25. It should be noted that several of the formulas in this section contain the number 3, which does not appear to be correct. It appears that the $\sum$ symbol would be more appropriate. In addition, the formula for RPD contains the symbol š where normally brackets would be used for the absolute value. This should be clarified and corrected.

10. **C1.1 Field Assessment and Response Actions**, page 1 of 5. It is stated that following completion of the field activities associated with each sampling event, the QA Officer will assess the work for the following items (a listing of six bulleted items is presented). It should be noted that assessments are best done throughout the project. It should be clarified what scope of authority the QA Officer has during the project, e.g. to intercede when quality is suspect during ongoing project operations, etc.

11. **D2.2 Laboratory Data Validation and Verification**, page 1 of 3.

a. It is stated in the first paragraph that the current USEPA Region 2 SOPs for SW-846 methods are available at http://www.epa.gov/region02/smb/sops.htm. It should be noted that the correct address is: http://www.epa.gov/region02/qa/documents.htm. In addition, SOP HW-22, Rev. 1, April 1995 is cited as current, however, the most recent SOP HW-22 is Rev. 2, June 2001. Also, a copy of the most recent cited HW-2, Rev. 13, September 2005, is attached, since it is not available at the listed website address. If any other required SOP is needed but not available at this site, please notify the EPA RPM.

b. It should be stated who will perform the data validation services, i.e., qualified Severn Trent laboratory QA personnel, personnel from the Technical Advisory Team, subcontractors, etc., and their appropriate qualifications.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION II

DATE: January 20, 2003

SUBJECT: Tracking Superfund Non-CLP Analytical Data (ANSETS): Directive # 9240.0-2C

FROM: Jennifer E. Feranda, CLP Project Officer and Regional Sample Control Coordinator
Hazardous Waste Support Section (2DESA-HWSB)

TO: See Addressees

The purpose of this memo is to inform you about OSWER Directive # 9240.0-2C (attached) concerning the requirements for nationally tracking non-Contract Laboratory Program (CLP) analytical services. This directive supersedes OSWER Directives 9240.0-2A and 2B which established the Analytical Services Tracing System (ANSETS). The primary focus of the new directive is on tracking analytical data generated via EPA field contractors and their subcontractors at federal fund lead sites and at sites where EPA is the lead agency overseeing federal facility cleanups under Interagency Agreements. Per the Directive, RS&T Division laboratories, State-funded sites, and Potentially Responsible Parties (PRPs) do not need to comply with ANSETS data submission. However, under the Region 2 non-CLP tracking system that was established in 2000, the Region requires PRPs to report this information. Site Project Managers should be writing this requirement into their action memos, orders, etc. Although this information is not currently required by HQ, the Region will continue to require PRPs to submit this data to the Regional Sample Control Coordinator (RSCC).

The EPA Field and Analytical Services Teaming Advisory Committee (FASTAC) established a decision tree for selecting analytical services. The Tiers (with Tier 1 being the most favorable option) are:

Tier 1: RS&T Division Laboratories (preferred option for special analytical services)
Tier 2: CLP (preferred option for routine analytical services)
Tier 3: Region specific analytical services contracts
Tier 4: Obtaining analytical services using subcontractors via field contracts.

Tier 4 is the least preferred option due to lack of direct oversight of these contractors, quality assurance potentially not meeting EPA standards, and often higher costs for services. By requiring contractors to use the ANSETS tracking system, the Superfund program can determine whether the FASTAC strategy is being implemented, analyze trends in new services needed, track national laboratory analyses acquired for the Superfund program, and plan for quality assurance oversight.

The directive outlines several options for submitting ANSETS data, however, it also allows each Region to use their discretion on how the ANSETS information will be provided to HQ. Region 2 already has a system in place for reporting Non-CLP analytical services information, and will continue to utilize this system. To provide consistency between the Region 2 and National requirements, the region will now utilize the "ANSETS Data Requirement" form in place of the "Non-CLP Tracking Form" which is currently being used. The forms should be completed and submitted to the RSCC, currently myself, by the first of each month for the previous months sampling. Once the information is compiled in the Region, it will be sent to HQ for inclusion in the national database.

The requirements for ANSETS reporting became effective as of January 1, 2003. Per the directive, Regional Contracting Officers and Project Officers will need to amend their assessment and response contracts to reinforce the ANSETS analytical services tracking requirements. If you have any questions regarding the implementation of these requirements, both national and regional, please contact me at (732) 321-6687.

Attachments

Addressees:

Shaheer Alvi
Helen Eng
Keith Moncino
Richard Graciano
Fernando Rosado
Kathy Moyik
Lisa Guarnieri
Superfund Remedial Project Managers
Superfund Site Assesment Managers
Superfund On-Scene Coordinators
HWSB

cc:
Vince Pitruzzello
Deb Szaro
Kevin Kubik

EPA/600/R
November 2002

Method TO-15 Supplement
Analysis of 1,1-DCE at pptv Concentrations

by

Karen D. Oliver, H. Herbert Jacumin, Jr., and E. Hunter Daughtrey, Jr.
ManTech Environmental Technology, Inc.
Research Triangle Park, NC 27709

and

W.A. McClenny
Human Exposure and Atmospheric Sciences Division
National Exposure Research Laboratory

National Exposure Research Laboratory
Office of Research and Development
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

Notice

The U.S. Environmental Protection Agency (EPA) through its Office of Research and Development (ORD), National Exposure Research Laboratory (NERL), is providing the information in this report as a result of funding received from the Regional Monitoring Initiative Program for applied research in support of the regional offices, especially Region 8, and in support of state programs, especially the Colorado Department of Public Health and Environment of the State of Colorado. A major portion of the report is the result of a work assignment under EPA Contract 68-D-00-206 to ManTech Environmental Technology, Inc. The report has been subjected to the Agency's peer and administrative review and has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

Abstract

The Supplement to EPA Compendium Method TO-15 provides guidance for reducing the method detection limit (MDL) for the compound 1,1-dichloroethene (1,1-DCE) and for other volatile organic compounds (VOCs) from 0.5 ppbv, as cited in Method TO-15, to much lower concentrations. Revisions to the original wording of Method TO-15 were made where the original language proved limiting to the goal of extending Method TO-15 to low pptv levels or where omissions or errors were observed. Also, recommendations in the form of additions were made on aspects of laboratory procedure deemed critical to low-pptv-level analysis. Specifically, the MDL for 1,1-DCE was determined to be 6 pptv. During this effort, a capability for preparing 1,1-DCE sample concentrations of 30 pptv and 60 ppbv in ambient air was developed. Using this capability and the capability to prepare samples of humidified zero air, samples were prepared in canisters and sent to three contract laboratories as unknowns. Subsequent comparison of results indicated close agreement among the laboratories while maintaining the performance standards for replicate precision (25%) and audit accuracy (30%) originally specified in Method TO-15. The following compounds were also detected at low pptv levels in canisters filled with spiked ambient air: chloroethene, dichloromethane, *cis*-1,2-dichloroethene, trichloromethane, 1,2-dichloroethane, benzene, 1,1,1-trichloroethane, trichloroethene, and tetrachloroethene. Since the different laboratories employed different analytical procedures, the use of a performance-based method appears justified. Specific guidance on analytical procedures from the Colorado Department of Public Health and Environment (CDPHE) is provided. These procedures have proven useful for CDPHE's contract laboratories in analyzing pptv-level samples of VOCs. The procedures followed by the EPA on-site contractor, ManTech Environmental Technology, Inc., in preparing and analyzing low-level concentrations of 1,1-DCE as well as other aspects of their work on this project are provided as Appendix A.

Foreword

The National Exposure Research Laboratory (NERL), Research Triangle Park, NC, performs research and development to characterize, predict, and diagnose human and ecosystem exposure, giving priority to that research which most significantly reduces the uncertainty in risk assessment and most improves the tools to assess and manage risk or to characterize compliance with regulations. The Laboratory seeks opportunities for research collaboration to integrate the work of the Office of Research and Development's (ORD) scientific partners and provides leadership to address emerging environmental issues and advance the science and technology essential for understanding human and ecosystem exposures. One aspect of the Laboratory's mission is to work with the U.S. Environmental Protection Agency's (EPA) regional and state offices.

EPA was asked by William P. Yellowtail, the Regional Administrator of Region 8, to review protocols from the Colorado Department of Public Health and Environment (CDPHE) for determining low parts per trillion by volume (pptv) concentrations of 1,1-dichloroethene (1,1-DCE) in ambient air in support of vapor intrusion monitoring. Tom Aalto of Region 8 coordinated the effort with EPA, and technical input on the CDPHE method was provided by Ken Niswonger and Edgar Ethington of CDPHE. The effort was funded under EPA's Regional Monitoring Initiative. In response to this request, NERL developed a work assignment for ManTech Environmental Technology, Inc., the current on-site contractor to NERL at the EPA facility in Research Triangle Park, NC. The task consisted of (1) developing a capability to support monitoring of 1,1-DCE at low-pptv concentrations at the EPA laboratory facilities, (2) documenting the existence of similar capabilities at representative contract laboratories, and (3) providing a TO-15 supplement that contains guidance for meeting the enhanced performance criteria.

Gary J. Foley, Ph.D.
Director
National Exposure Research Laboratory
Research Triangle Park, NC 27711

Contents

Tables

Acronyms and Abbreviations

µm	micrometer
1,1-DCE	1,1-dichloroethene
1,2-DCA	1,2-dichloroethane
CDPHE	Colorado Department of Public Health and Environment
CH_2Cl_2	dichloromethane
DQO	data quality objective
EM	electron multiplier
EPA	Environmental Protection Agency
FTDS	field test data sheet
L/min	liters per minute
MDL	method detection limit
mL/min	milliliters per minute
MS	mass spectrometry
NATA	National Air Toxics Assessment
NERL	National Exposure Research Laboratory
NIST	National Institute of Standards and Technology
ORD	Office of Research and Development
PFTBA	perfluorotributylamine
ppbv	parts per billion by volume
pptv	parts per trillion by volume
PQL	practical quantitation limit
RL	reporting limit
RRT	relative retention time
RSD	relative standard deviation
SIM	selected ion monitoring
TAMS	Toxics Air Monitoring System
TCE	trichloroethene
UATMP	Urban Air Toxics Monitoring Program
VOC	volatile organic compound
°C	degrees Celsius

Acknowledgments

The authors thank Tom Aalto of Region 8 for his assistance in starting this research and the scientists at the Colorado Department of Public Health and Environment (CDPHE), particularly Ken Niswonger and Edgar Ethington, for providing their input in the form of Appendix B to this report. Also, thanks to Bill Lonneman, an experienced senior scientist now working with the U.S. Environmental Protection Agency (EPA) as part of the Senior Environmental Employment program, for providing advice on analytical procedures and helping with the substantiation of target compound concentration levels in gas standards. The authors would like to acknowledge the efforts of Stacy Henkle of KulTech, Inc., in editing and formatting this document.

Chapter 1
Introduction

This document is a supplement to Method TO-15 in the EPA Compendium of Methods for Air Toxics. It addresses the use of specially prepared canisters for monitoring a single, specific chlorinated compound, 1,1-dichloroethene (1,1-DCE), with implications for the monitoring of other compounds and for multiple compounds in samples containing compound mixtures. Recent reevaluation of risk levels specifically for 1,1-DCE indicates a lowered risk level compared to that established earlier. However, the guidance presented in this document remains relevant to other compounds for which 1,1-DCE can be considered a surrogate.

TO-15 is a performance-based method prepared by EPA as a guidance document for monitoring subsets of those volatile organic compounds (VOCs) that are mentioned in Title III of the Clean Air Act Amendments of 1990. The TO-15 performance criteria are based on data from existing databases compiled in national monitoring programs (e.g., the Toxics Air Monitoring System [TAMS] and Urban Air Toxics Monitoring Program [UATMP]) using canister-based sampling and bench-top quadrupole mass spectrometers. These performance criteria provide a method detection limit (MDL), a method replicate precision, and a method audit accuracy. The sampling and analytical approaches are not restricted in any sense as long as the performance criteria are met. Examples of possible approaches to analysis, generation of calibration mixtures, and use of quality control measures (technical acceptance criteria) are provided in the text of TO-15. These examples are intended to be instructive, not prescriptive.

The TO-15 Supplement is currently restricted to canister-based systems for monitoring target compound concentrations lower than the 0.5 parts per billion by volume (ppbv) stated as one of the TO-15 performance criteria. This enhancement of monitoring capability is typically required if monitoring at 10^{-6} risk levels of high-risk compounds must be done. These levels can be quite low as noted in Table 1, which lists the cancer risk

Table 1. Risk Levels for NATA Compounds (from www.epa.gov/iris/)

#	TO-14 Compounds	TO-14 #	NATA List	E-6 (1 in 1,000,000) Risk Level mg/m³	Molecular Weight	Risk Level ppbv
1	Vinyl chloride	4	Yes	$2.3x10^{-4}$	62.50	90.0
2	1,1-Dichloroethene*	8		$2x10^{-5}$	96.94	5.0
3	Dichloromethane	9	Yes	$2x10^{-3}$	84.93	575.8
4	Trichloromethane	14	Yes	$4x10^{-5}$	119.38	8.2
5	1,2-Dichloroethane	15	Yes	$4x10^{-5}$	98.96	9.9
6	Benzene	17	Yes	$1.3x10^{-4}$	78.12	40.7
7	Carbon tetrachloride	18	Yes	$7x10^{-5}$	153.82	11.1
8	1,2-Dichloropropane	19	Yes	Not established	112.99	Not established
9	Trichloroethene	20	Yes	Not established	131.29	Not established
10	*cis*-1,3-Dichloropropene	21	Yes	$2x10^{-4}$	110.97	44.1
11	*trans*-1,3-Dichloropropene	22	Yes	$2x10^{-4}$	110.97	44.1
12	1,1,2-Trichloroethane	23		$6x10^{-5}$	133.41	11.0
13	1,2-Dibromoethane	25	Yes	$5x10^{-6}$	187.87	0.7
14	Tetrachloroethene	26	Yes	Not established	165.83	Not established
15	1,1,2,2-Tetrachloroethane	31	Yes	$2x10^{-5}$	167.85	2.9
16	Hexachlorobutadiene	41		$5x10^{-5}$	260.76	4.7
#	Other Compounds	TO-14 #	NATA List	E-6 (1 in 1,000,000) Risk Level mg/m³	Molecular Weight	Risk Level ppbv
17	Acrylonitrile		Yes	$1x10^{-5}$	53.06	4.6
18	1,3-Butadiene		Yes	$4x10^{-6}$	54.09	1.8
19	Ethylene oxide		Yes	Not established	44.05	Not established

*Risk level prior to reevaluation.

levels of the National Air Toxics Assessment (NATA) compounds. The approach taken in the Supplement is to retain the performance criteria of precision and accuracy while reducing the MDLs to meet more stringent data quality objectives (DQOs). An example of an analytical approach taken by CDPHE is presented along with an analytical approach taken by EPA in-house contractor ManTech Environmental Technology, Inc. CDPHE has developed a set of specifications that are used in purchasing analytical services from contract laboratories. These analytical specifications provide practical guidance in achieving the enhanced performance required for high-risk compounds.

EPA, through its in-house contractor, prepared a set of canisters filled with various levels of 1,1-DCE in a mixture and as a single compound in ambient air, as well as canisters filled with humidified zero air. These samples have been analyzed by four laboratories to obtain an idea of the agreement expected and to verify that low concentration levels corresponding to 10^{-6} risk levels can actually be quantified. While these tests provide an example of how well such samples are likely to be analyzed, it does not mean that other laboratories would do better or worse, or that a non-canister approach to sampling would not do as well or better.

In summary, the Supplement acknowledges the need for sampling and analytical protocols that reduce the MDLs for certain types of measurements and provides examples of achieving this reduction. The analytical guidelines developed by CDPHE for use by their contract laboratories, for example, provide a useful and practical approach for current monitoring applications. The agreement among the four laboratories (see Appendix A) establishes that more than one analytical approach is viable and, furthermore, that the preparation of canisters and standards for sampling 1,1-DCE is possible at low parts per trillion by volume (pptv) levels. The extension to other single compounds and to multiple compounds should be straightforward.

Chapter 2
Conclusions

1. The TO-15 Supplement provides guidance for sampling and analysis of 1,1-DCE, and by implication other VOCs, in air at levels lower than the TO-15 MDL of 0.5 ppbv, with the specific level depending on the DQOs for the project at hand. The performance criteria are an MDL at the customized DQO levels, replicate precision of at least 25%, and audit accuracy of 30%.

2. The Supplement includes revisions and additions by section to the original TO-15 Method. As an addition to section 10.2.3, two examples of technical approaches to meet the performance criteria are provided. One is the guidance developed during this project by EPA on-site contractor ManTech Environmental Technology, Inc. (Appendix A); the other is a concise restatement of the guidance developed by CDPHE for the analysis of high-risk compounds associated with vapor intrusion (Appendix B).

3. Samples of 30 and 60 pptv of 1,1-DCE in ambient air prepared by ManTech Environmental Technology, Inc., were analyzed by four laboratories, and the results showed that the TO-15 Supplement performance criteria could be met at concentrations as low as 30 pptv. One of the laboratories was the EPA on-site laboratory operated by ManTech, and at least one of the other contract laboratories used the CDPHE guidance.

Chapter 3
Recommendations

1. The technical acceptance criteria provided in the original TO-15 and in the TO-15 Supplement must be recognized as guidance. Other technical acceptance criteria can be used for meeting the performance criteria of TO-15 and the TO-15 Supplement. This point is evidenced by the close agreement of results obtained by four independent laboratories analyzing identical samples, each using their own standard operating procedures.

2. Laboratories wishing to perform analyses of VOCs at low-pptv levels must exercise diligence in all aspects related to cleanliness (canister cleanup and certification, carryover issues, instrument background levels, etc.). In addition, accurate calibration standards at the appropriate concentrations must be obtained or generated. Finally, the MS method will need to be optimized according to the specific analytical system used and the analyte(s) chosen.

3. Agreement on the audit standards to be used in monitoring low-level VOCs is necessary whether the audit standard is to be the average of analysis results from different laboratories, diluted National Institute of Standards and Technology (NIST)–traceable standards from commercial suppliers, or fundamentally derived standards. For the TO-15 recommendations see section 9.2.

4. Caution should be exercised when working at low-pptv levels due in part to the need for a more rigorous investigation of storage stability and sample integrity issues as well as a general need for more laboratory tests in the low-pptv range of sample concentrations. Extreme conditions of humidity (<15% RH for any sample and high humidity for positive pressure samples) and of co-collected reactive compounds may complicate the sampling and analytical conditions. More experience is needed in monitoring at low-pptv levels.

5. To confirm consistent sampling technique, a number of replicate samples should be collected and analyzed.

Chapter 4
Method TO-15 Supplement
Correspondence to TO-15 Section Numbers

The Method TO-15 supplementary material that applies to the determination of 1,1-DCE at low-pptv concentrations is enumerated below by Method TO-15 section number. Each section is labeled as either a revision or an addition. When a revision is noted, the italicized text is the text that has been revised. The supplementary material is presented in this format to provide clarity for the reader by consolidating the relevant Method TO-15 sections into a concise text.

1.2 [REVISION] This method applies to *low-pptv-level* ambient concentrations of 1,1-DCE and typically requires VOC enrichment by concentrating up to one liter of a sample volume. The VOC concentration range for ambient air in many cases includes the concentration at which continuous exposure over a lifetime is estimated to constitute a 10^{-6} or higher lifetime risk of developing cancer in humans. Under circumstances in which many hazardous VOCs are present at 10^{-6} risk concentrations, the total risk may be significantly greater.

3.1 (last bullet) [REVISION]

- Finally, Compendium Method TO-15 includes enhanced provisions for inherent quality control. *Recommendations for the method include* internal analytical standards and frequent verification of analytical system performance to assure control of the analytical system. This more formal and better documented approach to quality control *should result in* a higher percentage of good data.

6.2.4 [REVISION] Significant contamination of the analytical equipment can occur whenever samples containing high VOC concentrations are analyzed. This in turn can result in carryover contamination in subsequent analyses. Whenever a high concentration *(a suggested rule of thumb is 100 times the MDL)* sample is encountered, it should be followed by an analysis of humid zero air to check for carryover contamination.

6.2.5 [REVISION] In cases when solid sorbents are used to concentrate the sample prior to analysis, the sorbents should be tested to identify artifact formation (see Compendium Method TO-17 for more information on artifacts *and sorbents*).

7.3.3 Electronic Mass Flow Controllers. [REVISION] One 0 to 5 L/min unit *for air* and one or more *0 to 10 mL/min or* 0 to 100 mL/min units for *nitrogen (standard cylinder make-up gas)*, depending on the number of cylinders in use for calibration and the dilution requirements.

8.3.5 [REVISION] To verify correct sample flow, a "practice" (evacuated) canister is used in the sampling system.

[**Note:** For a subatmospheric sampler, a flow meter and practice canister are needed. For the pump-driven system, the practice canister is not needed, as the flow can be measured at the outlet of the system.]

A certified mass flow meter is attached to the inlet line of the manifold, just in front of the filter. The canister is opened. The sampler is turned on and the reading of the certified mass flow meter is *observed.* The value should *be* within ±10% *of the correct value*. If not, the sampler mass flow *controller control unit should be adjusted to give the correct sample flow rate. If an unusually large adjustment of the mass flow controller control unit is necessary to obtain the correct flow, then other problems such as leaks in the system should be investigated and corrected.*

[**Note:** Mass flow meter readings may drift. Check the zero reading carefully and add or subtract the zero reading when reading or adjusting the sampler flow rate to compensate for any zero drift.]

Record final flow under "CANISTER FLOW RATE" on the field test data sheet (FTDS).

8.4 Cleaning and Certification Program [ADDITION]

- Extremely clean and leak-free canisters are key to meeting the TO-15 acceptance criteria at low-pptv levels.
- Temperatures at or above 100 °C, when combined with alternating high vacuum and humidified ultra-clean air purges, are an excellent way to remove contaminants from the canister. Commercially manufactured canister cleaners which incorporate all these features are currently available.

[**Note:** Check with the manufacturer of the canister valve for information on the temperature limits of the valve so as to prevent any damage to the valve.]

- Canisters known to contain high levels of contaminants may be alternately "rough pumped" to moderate vacuum and vented to ambient pressure under a hood for several cycles before the canisters are placed in the cleaning system if there is a possibility that the canister cleaning system itself might become contaminated by the high levels of contaminants in the canisters.
- Canisters filled with humidified ultra-clean air and awaiting cleanliness certification should be allowed to "age" or equilibrate for a minimum of 24 hours, with several days being recommended.
- A canister should be considered clean if the analysis of humidified ultra-clean air reveals no target VOCs above the MDL for those target VOCs. The number of cleaning cycles required to achieve this stringent goal will vary depending on the type and concentration of analytes previously sampled and on the capabilities of the particular cleaning system used.
- A canister should be considered relatively leak-free if after being evacuated to <25 μm Hg, there is a 20 μm Hg or less increase in pressure after a 24-hour or longer period. However, to eliminate the possibility of contamination of cleaned canisters by influx of ambient air prior to sampling, a "zero tolerance" leak policy is recommended.

9.2 Preparation of Standards [ADDITION]

- The concentration of a primary standard chosen by a laboratory should be based on the ability of that laboratory to consistently and accurately reproduce working calibration standards over the specific calibration range of that laboratory.
- Certain vendors now offer cylinder standards for specific VOCs at 10-ppb levels and TO-14 mixtures as low as 100 ppbv. Primary standards at these concentrations allow preparation of working standards at the low-pptv level.

[**Note:** Pay close attention to the linear range of the mass flow controllers used to create the standard dilutions.]

9.2.2.2 Calibration Standards [ADDITION]

- Working calibration standards should be prepared in a range of concentrations that reflect the entire reporting range for the analytes of interest.

10.2.3 [ADDITION 1 – Suggestions for optimization of a mass spectrometry scan method for detection at low-pptv levels]

- Based on the molecular weight of the VOC or VOCs of interest, the scan range for the mass spectrometry (MS) method may be narrowed. For ion trap instruments in particular, the background mass as well as the segment radio-frequency value and the automatic gain control prescan storage level may be increased.
- The scan time for the MS method may be reduced in order to provide better resolution of peaks. However, most systems will have a minimum scan time threshold below which sensitivity decreases in response to shorter scan times. Optimization is the key word.
- The above adjustments to a working MS method to enhance sensitivity (i.e., lower the MDL) should only be implemented after a thorough investigation of their individual and collective effects on system response to the target analytes.
- An example of an enhanced MS scan method for the specific detection of 1,1-DCE at low-pptv levels is given in Appendix A of this report.

[ADDITION 2 – Suggestions for optimization of MS–selected ion monitoring (SIM) and MS scan methods for detection at low-pptv levels]

- An example of guidance developed by CDPHE for use in the analysis of canister-based samples for high-risk VOCs is given in Appendix B.

10.5 Initial Calibration [ADDITION]

- If the analytical range of interest is 20 to 500 pptv, then the five calibration concentrations chosen might be 10, 25, 50, 200, and 500 pptv. For calibrations over a large range, more than five calibration concentrations may be selected.

[**Note:** Levels as high as or higher than 500 pptv may present carryover problems in some systems for subsequent analyses at low-pptv levels. It is suggested that a humidified ultra-clean air blank be run following any analysis in which the level of any target analyte is 100 times its MDL or greater.]

- One of the calibration points from the initial calibration curve should be at the same concentration as the daily calibration standard (e.g., 50 pptv).

10.6 Daily Calibration [ADDITION]

- The daily calibration standard (e.g., 50 pptv) should contain all the target compounds.

10.7 Blank Analyses [ADDITION]

- The sorbents used in sorbent preconcentrators will often introduce a background into each analysis upon thermal desorption. It is also possible that there could be a certain amount of outgassing from components in the analytical system. When running ppbv-level analyses, these levels should be negligible. However, at low-pptv levels this background contamination can become significant. It is important to characterize this system background through identification and quantification of the specific contaminants. This could be accomplished by analyzing a series of 10 canisters filled with humidified ultra-clean air or by 10 consecutive analyses of humidified ultra-clean air from a continuously purged clean manifold. The results would be compiled into a spreadsheet and any outliers (high results implying a true background in the canister) could be eliminated. From the remaining results an average background level for each contaminant could be calculated. This background level for each contaminant could then be subtracted from the analytical results of actual samples in order to provide the most accurate data.

- A daily laboratory method blank should still be run as a way to check for any new contamination possibly introduced through the sample analysis process. In addition, the daily laboratory method blank acts as a humid purge of the analytical system.

11.1.3 [ADDITION] The recommendation for extending the MDL for 1,1-DCE to a low-pptv concentration while maintaining the standard Method TO-15 requirements for replicate precision and audit accuracy is based on an EPA-sponsored assessment of the capabilities of laboratories that are currently analyzing ambient air samples containing 1,1-DCE at pptv concentrations. The data supporting replicate precision within 25% have been extracted from the Appendix A report and reproduced for the reader's convenience in Tables 2 through 5. As a measure of audit precision, the excellent agreement among four laboratories analyzing replicate samples of ambient air containing low-pptv levels of 1,1-DCE (Tables 2–5) was considered. For the 30-pptv samples (Tables 2 and 3), the relative standard deviation (RSD) of 18 measurements was 5.5%. For the 60-pptv samples (Tables 4 and 5), the RSD of 12 measurements was 6.8%. Obviously, there exists a need in the VOC analysis community for a NIST-certified gaseous audit standard of 1,1-DCE at low-pptv concentrations so that a true audit may be conducted to ensure that a laboratory meets the Method TO-15 performance criteria for audit accuracy. Despite the fact that a true audit could not be conducted, the agreement among the four laboratories, each of which used varying approaches to instrument calibration, is a measure of audit accuracy. Taking the mean of all measurements made by the laboratories as the "true" concentration, the percent differences between each of the measurements made by the four laboratories and the "true" concentration for 1,1-DCE ranged from 0 to 17%.

Table 2. Low-Level Method TO-15 SIM Analytical Results for Ambient Air Spiked with 1,1-DCE (results in pptv)

CANISTER	A-701	785	GA-B	RSD	RL	120	01578	MTC-22	RSD	RL	208	013	454	RSD	PQL
Compound LAB	1	1	1		1	2	2	2		2	3	3	3		3
1,1-Dichloroethene	30	30	30	0.0	10	28	29	27	3.6	10	27	29	29	4.2	10

RSD = relative standard deviation
RL = reporting limit
PQL = practical quantitation limit

Table 3. Low-Level Method TO-15 Scan Analytical Results for Ambient Air Spiked with 1,1-DCE (results in pptv)

CANISTER	A-701	785	GA-B	120	01578	MTC-22	208	013	454	RSD	PQL
Compound LAB	4	4	4	4	4	4	4	4	4		4
1,1-Dichloroethene	32	27	30	30	32	29	32	31	29	5.7	18

RSD = relative standard deviation
PQL = practical quantitation limit

Table 4. Low-Level Method TO-15 SIM Analytical Results for Ambient Air Spiked with a Chlorinated Gas Mixture Containing 1,1-DCE (results in pptv)

CANISTER	N-3	726	Percent Difference	RL	096	727	Percent Difference	RL	9682-B	9677-B	Percent Difference	PQL
Compound LAB	1	1		1	2	2		2	3	3		3
1,1-Dichloroethene	59	59	0.0	10	60	53	12.4	10	60	54	10.5	10

RL = reporting limit
PQL = practical quantitation limit

Table 5. Low-Level Method TO-15 Scan Analytical Results for Ambient Air Spiked with a Chlorinated Gas Mixture Containing 1,1-DCE (results in pptv)

CANISTER	N-3	726	096	727	9682-B	9677-B	RSD	PQL
Compound LAB	4	4	4	4	4	4		4
1,1-Dichloroethene	69	59	60	61	57	62	6.7	18

RSD = relative standard deviation
PQL = practical quantitation limit

Appendix A
Determination of Low-pptv Concentrations of 1,1-Dichloroethene in Ambient Air Collected in Specially Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)

by

Karen D. Oliver, H. Herbert Jacumin, Jr., and E. Hunter Daughtrey, Jr.
ManTech Environmental Technology, Inc.
Research Triangle Park, NC 27709

and

W.A. McClenny
Human Exposure and Atmospheric Sciences Division
National Exposure Research Laboratory

Abstract

An optimized mass spectrometry (MS) scan method was developed in this laboratory for the detection of 1,1-dichloroethene (1,1-DCE) at low parts per trillion by volume (pptv) levels. A cylinder standard of 10 parts per billion (ppb) 1,1-DCE in nitrogen was purchased from a commercial vendor and is used for the preparation of canister standards of 1,1-DCE at concentrations as low as 2.5 pptv. A method detection limit (MDL) of 6 pptv was determined for 1,1-DCE using the optimized scan method, and linearity of detector response over a range of 10 to 200 pptv was demonstrated. Next, an experiment was designed to assess the capabilities of several contract laboratories that currently offer low-level Method TO-15 type analyses of 1,1-DCE and other volatile organic carbons (VOCs) to their clients. Despite differences in instrumentation, MS mode of operation (SIM or scan), MS tuning methods, and calibration standards and techniques used by the four laboratories, excellent agreement was achieved for the determination of 1,1-DCE at nominal concentrations of 30 and 60 pptv in canister samples of spiked ambient air. The excellent agreement for 1,1-DCE is indicated by relative standard deviations of replicate measurements of ≤7%, computed for experiments in which three to 18 measurements were available. Replicate precision results (calculated as percent difference) for those experiments in which two samples were analyzed by each individual contract laboratory were <13% for 1,1-DCE.

The excellent agreement in analytical results for the four laboratories that analyzed canister samples of ambient air containing 1,1-DCE at 30 and 60 pptv demonstrates that Method TO-15 has been successfully extended to low-pptv concentrations of analytes. MDLs of 0.5–6 pptv and reporting/quantitation limits of 10–20 pptv have been achieved. Additionally, since the four laboratories used different approaches for the low-level Method TO-15 analyses, the results support the premise of a performance-based methodology that focuses on MDLs, audit accuracy within 30%, and replicate precision within 25% as indicators of method acceptability.

Preface

The EPA was asked by William P. Yellowtail, the Regional Administrator of Region 8, to review protocols from the Colorado Department of Public Health and Environment (CDPHE) for determining low-pptv concentrations of 1,1-dichloroethene (1,1-DCE) in ambient air in support of vapor intrusion monitoring. Tom Aalto of Region 8 coordinated the effort with EPA, and technical input on the CDPHE method was provided by Ken Niswonger and Edgar Ethington of CDPHE. The effort was funded under EPA's Regional Monitoring Initiative. In response to this request, NERL, EPA developed a work assignment to ManTech Environmental Technology, Inc., the current on-site contractor to the National Exposure Research Laboratory (NERL) at the EPA facility in Research Triangle Park, NC. The task consisted of: (1) developing a capability to support monitoring of 1,1-DCE at low-pptv concentrations at the EPA laboratory facilities; (2) documenting the existence of similar capabilities at representative contract laboratories; and (3) providing a TO-15 supplement that contains guidance for meeting the enhanced performance criteria.

Foreword

This technical report presents the results of work performed by ManTech Environmental Technology, Inc., under Contract 68-D-00-206 for the Human Exposure and Atmospheric Sciences Division, National Exposure Research Laboratory, U.S. Environmental Protection Agency (EPA), Research Triangle Park, NC. This technical report has been reviewed by ManTech Environmental Technology, Inc., and approved for publication. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

Contents

Figures

Tables

Acronyms and Abbreviations

%D	percent difference
µm	micrometer
1,1-DCE	1,1-dichloroethene
AGC	automatic gain control
amu	atomic mass units
autoGC-MS	automated gas chromatograph/mass spectrometer
cc	cubic centimeter
CDPHE	Colorado Department of Public Health and Environment
CV	coefficient of variation
ECD	electron capture detector
EPA	U.S. Environmental Protection Agency
FID	flame ionization detection
g	gram
GC	gas chromatography
h	hour
HSA	humidified scientific-grade air
i.d.	inside diameter
LMB	laboratory method blank
MDL	method detection limit
m	meter
mm	millimeter
MS	mass spectrometry
m/z	mass to charge ratio
NIST	National Institute of Standards and Technology
PAMS	Photochemical Assessment Monitoring Stations
ppbv	parts per billion by volume
pptv	parts per trillion by volume
PQL	practical quantitation limit
psig	pounds per square inch gauge
RF	radio frequency
RH	relative humidity
RL	reporting limit
RSD	relative standard deviation
sccm	standard cubic centimeters per minute
SD	standard deviation
SIM	selected ion monitoring
SRM	Standard Reference Material
VOCs	volatile organic compounds
°C	degrees Celsius

Chapter 1
Introduction

The U.S. Environmental Protection Agency (EPA) Method TO-15 is titled "Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)" and is a part of the EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air. It is a performance-based method consisting of three performance criteria and guidance (including a suggested set of technical acceptance criteria) to verify analytical system control in order to meet the performance criteria.

In the current effort, Method TO-15 has been modified to reduce the method detection limits (MDLs) while maintaining the specifications for replicate precision and audit accuracy. Suggestions for technical approaches that will enhance analytical system performance so as to meet replicate precision and audit accuracy at the lowered MDLs have been provided in the supplement. In addition, guidance on sampling using canister-based systems is provided. Finally, the achievement of the performance criteria at representative laboratories has been demonstrated.

This report documents the experimental evidence that is the basis for the supplement to Method TO-15 and is complementary to the supplement. The essential result of this report and the Method TO-15 Supplement is a modified Method TO-15 for determination of parts per trillion by volume (pptv) concentrations of 1,1-dichloroethene (1,1-DCE) (and by implication other VOCs) and the verification that laboratories using different technical acceptance criteria can meet a set of performance criteria consisting of pptv MDLs while retaining the replicate precision and audit accuracy requirements of Method TO-15.

Chapter 2
Conclusions

An optimized MS scan method was developed in this laboratory for the detection of 1,1-DCE at low-pptv levels. A cylinder standard of 10 parts per billion (ppb) 1,1-DCE in nitrogen was purchased from a commercial vendor and is used for the preparation of canister standards of 1,1-DCE at concentrations as low as 2.5 pptv. An MDL of 6 pptv was determined for 1,1-DCE using the optimized scan method, and linearity of detector response over a range of 10 to 200 pptv was demonstrated. An informal storage stability study for low-pptv concentrations of 1,1-DCE in air samples collected and stored in canisters manufactured by several vendors was conducted.

An experiment was designed to assess the capabilities of several contract laboratories that currently offer low-level Method TO-15 type analyses of 1,1-DCE and other VOCs to their clients. Despite differences in instrumentation, MS mode of operation (selected ion monitoring [SIM] or scan), MS tuning methods, and calibration standards and techniques used by the four laboratories, excellent agreement was achieved for the determination of 1,1-DCE at nominal concentrations of 30 and 60 pptv in canister samples of spiked ambient air. The excellent agreement for 1,1-DCE is indicated by relative standard deviations (RSDs) of replicate measurements of ≤7%, computed for experiments in which three to 18 measurements were available. The RSDs of three replicate quantitative measurements of the additional VOCs (dichloromethane, trichloromethane, 1,1,1-trichloroethane, benzene, and tetrachloroethene) measured by each individual contract laboratory were <12%. Replicate precision results (calculated as percent difference) for those experiments in which two samples were analyzed by each individual contract laboratory were <13% for 1,1-DCE and <15% for 17 of 20 comparisons for the additional VOCs (chloroethene, dichloromethane, *cis*-1,2-dichloroethene, trichloromethane, 1,2-dichloroethane, 1,1,1-trichloroethane, benzene, trichloroethene, and tetrachloroethene). The analytical results for 1,1-DCE from the contract laboratories showed better agreement for the low-level Method TO-15 type analyses than they did for the Method TO-14 analyses of canister samples containing 60 VOCs at nominal concentrations of 5 parts per billion by volume (ppbv).

The excellent agreement in analytical results for the four laboratories that analyzed canister samples of ambient air containing 1,1-DCE at 30 and 60 pptv demonstrates that Method TO-15 has been successfully extended to low-pptv concentrations of analytes. MDLs of 0.5–6 pptv and reporting/quantitation limits of 10–20 pptv have been achieved. Additionally, since the four laboratories used different approaches for the low-level Method TO-15 analyses, the results support the premise of a performance-based methodology that focuses on MDLs, audit accuracy within 30%, and replicate precision within 25% as indicators of method acceptability.

Chapter 3
Recommendations

Recommendations for further sample integrity studies and the need for gaseous audit standards of VOCs at pptv concentrations are discussed below. In addition, practical advice is offered in several areas of importance for sampling and analysis of pptv concentrations of 1,1-DCE and other VOCs.

(1) Further sample integrity studies are needed in the following areas:

- *Storage stability studies.* A well-controlled storage stability study for samples of ambient air containing pptv concentrations of 1,1-DCE and additional VOCs that are collected and stored in canisters is needed. The experiments conducted for this report were informal "before and after" type experiments for a small number of samples. A more thorough experiment in which a statistically significant number of samples of ambient air containing pptv-level VOCs stored in canisters are analyzed on days 0, 2, 4, 7, 15, and 30 is needed. The experiment should be designed to include canisters from various vendors.

- *Investigation of initial losses.* A sample integrity study to investigate the possibility of initial losses of pptv concentrations of VOCs to the canister walls is needed. In the experiments discussed in this report, a small difference was observed in the real-time spiked ambient air measurements for 1,1-DCE that were made with the EPA autoGC/MS system using the optimized Method TO-15 scan method while the canister samples were being prepared as compared to the later measurements for the canister samples. For the samples of ambient air spiked with 1,1-DCE, the mean concentration of 1,1-DCE was 33 pptv for the real-time measurements versus 30 pptv for the canister measurements. For the samples of ambient air spiked with a mixture of chlorinated VOCs, the mean concentration of 1,1-DCE was 68 pptv for the real-time measurements versus 62 pptv for the canister measurements. Additional experiments are needed to investigate this difference between the real-time and canister measurements.

(2) A gaseous audit standard of pptv concentrations of 1,1-DCE and other VOCs is needed to determine whether a laboratory can meet the Method TO-15 performance criteria for audit accuracy. For this report, the results of analyses of spiked ambient air samples by four laboratories are used as a measure of audit accuracy. However, a National Institute of Standards and Technology (NIST)–certified audit standard is needed to evaluate those laboratories who analyze VOCs according to the Method TO-15 Supplement for analysis of low-pptv concentrations of 1,1-DCE.

(3) Practical advice for sampling and analysis of pptv concentrations of 1,1-DCE and other VOCs is offered here as a service to the reader:

- As expected, the preparation and analysis of air samples containing pptv levels of VOCs requires that the analytical and standards preparation systems be extremely clean. Laboratory personnel must take great care to ensure that the sample preparation system and/or analytical system is thoroughly purged with humidified air or nitrogen after higher concentrations of VOCs have been present in the systems and prior to preparation and/or analysis of low-pptv-level VOCs. Verification of cleanliness with a laboratory method blank (LMB) of humidified air or nitrogen is needed even if the samples that were prepared or analyzed previously contained VOCs at low-ppbv concentrations. Humidity is an essential factor in the cleanliness verification process because analysis of a dry sample does not always yield an accurate characterization of a system's cleanliness.[1,2] An example of this is seen in this laboratory with the EPA autoGC/MS system in which analysis of a helium blank sample shows the system to be free of artifact peaks whereas analysis of a sample of humidified air results

in the detection of artifact peaks such as benzene that are associated with the use of the multisorbent traps.

- Particular care must be given to canister cleanliness when samples containing pptv concentrations of 1,1-DCE and other VOCs are collected and analyzed. Once a laboratory has established an MDL and a practical quantitation limit (PQL) for an analyte such as 1,1-DCE, the sampling canisters should be subjected to a blank certification process to ensure that the analyte is not present in the canister at a concentration that is greater than the MDL for that analyte. The blank certification process should be conducted by filling a clean canister with humidified air or nitrogen and allowing the canister to "age" for a minimum of 24 hours prior to analysis. In the experiments conducted for this report, canisters in this laboratory that had previously been used for multicomponent VOC standards at concentrations as great as 40 ppbv were successfully cleaned and used for analysis of 1,1-DCE at concentrations as low as 5 pptv. Additionally, the results of the canister cleanliness tests that were conducted on canisters rented from the contract laboratories showed that the canisters generally were clean with respect to the target compounds. However, a non-target compound (toluene) was observed in one canister at 714 pptv, a concentration that is greater than that specified under Method TO-14 and TO-15 technical acceptance criteria. As an additional measure of quality control with respect to canister cleanliness, laboratory staff may wish to designate that specific canisters be reserved for use with samples containing ultra-trace-level concentrations of specific VOCs.

- While conducting the experiments discussed in this report, laboratory staff noted that a greater amount of time was required for thorough equilibration of the analytical sampling train when standards containing pptv concentrations of VOCs were analyzed as compared to the equilibration time required for standards containing ppbv concentrations of VOCs. Obviously, this observation is system specific and is dependent on both the length of tubing in the analytical system and the complexity of the individual analytical system. The issue of equilibration is mentioned here simply to generate awareness of a potential problem.

Chapter 4
Experimental Method and Results

Under contract to EPA, ManTech Environmental Technology, Inc., was given the task of evaluating the ability of several contract laboratories to determine 1,1-DCE at low-pptv concentrations in air samples. The results of this evaluation would be used to determine whether EPA Method TO-15 could be extended successfully to the analysis of low-pptv concentrations of 1,1-DCE. In response to this task, ManTech developed a work plan that called for (1) developing the capability of preparing pptv-level calibration standards for 1,1-DCE; (2) refining our current TO-15 GC/MS method for the detection of 1,1-DCE at pptv levels; (3) evaluating the cleanliness of our canisters and analytical system for monitoring pptv concentrations of 1,1-DCE; (4) determining the storage stability of 1,1-DCE at pptv levels in air samples collected and stored in canisters; and (5) evaluating the capabilities of contract laboratories that are already providing commercial analyses of selected chlorinated VOCs, including 1,1-DCE, at low-pptv levels for their clients.

4.1 Calibration Standard

Preparation of calibration standards of VOCs at concentrations of 0.5 to 100 ppbv has been accomplished in this laboratory by dynamic dilution of 2 to 10 parts per million concentrations of VOCs in high-pressure cylinders using scientific-grade air (National Specialty Gases, Durham, NC) and mass flow controllers.[3] In order to use this same technique for preparation of low-pptv concentrations of 1,1-DCE, a 10-ppb standard of 1,1-DCE in nitrogen in a high-pressure cylinder was purchased from Spectra Gases Inc. (Branchburg, NJ). An analysis value of 10 ppb was reported by Spectra Gases for 1,1-DCE. Dynamic dilution of the 10-ppb standard of 1,1-DCE allows for the preparation of calibration standards at concentrations as low as 2.5 pptv.

The cylinder concentration of 10 ppb for 1,1-DCE was verified by analysis in an independent laboratory within EPA. In that laboratory, a GC with flame ionization detection (FID) per carbon approach is employed and the instrument calibration is based on a NIST/Standard Reference Material (SRM) propane in air cylinder.[4] The concentration of the standard sampled directly from the cylinder in February 2002 was determined by the GC-FID method to be 10.10 ppbv ±0.98% coefficient of variation (CV). The cylinder was reanalyzed in June 2002, and the concentration of 1,1-DCE was determined to be 10.23 ppbv ±1.24% CV. In September 2002, a third analysis of the 1,1-DCE standard was performed in the same GC-FID laboratory. For this analysis, the standard was provided in a canister that had been prepared by injecting 90 µL of water into the evacuated canister and then pressurizing the canister with the gas standard directly from the cylinder. The GC-FID analytical result for the canister standard was 10.52 ppbv ±0.58% CV based on the NIST/SRM propane standard calibration.

For the experimental work discussed in this report, the nominal value of 10 ppb was used for the standard cylinder to calculate concentrations of 1,1-DCE canister standards prepared by dynamic dilution.

4.2 Method Development

4.2.1 Optimization of the MS Scan Method

An autoGC/MS system was operated in this laboratory to determine VOCs in whole air samples using a modified Method TO-15 approach.[5] The autoGC/MS system consisted of a XonTech 930 organic vapor concentrator, which uses two multisorbent traps and a XonTech 940 cryogenic concentrator (RM Environmental Systems, Inc., Van Nuys, CA), interfaced to a Varian 3800 GC and Saturn 2000 ion trap MS (Varian Inc., Walnut Creek, CA). The multisorbent traps contained 0.05 g of Tenax GR, 0.04 g of Carbotrap, and 0.51 g of Carbosieve S III. A total sample volume of 285 cc was collected; however, a 4:1 split at the head of the column reduced the sample volume that was routed to the detector from 285 to 57 cc. A 60 m by 0.32 mm by 1.0 µm DB-1 capillary column (Agilent Technologies, Wilmington, DE) was used for separation of analytes. The GC oven temperature was programmed as follows: 35 °C for 5 min, a 6 °C/min ramp to 210 °C, and a 0.84-min hold at 210 °C, for a total analysis time of 35 min. The standard MS operating parameters are listed in Table 4-1. In the Saturn software, quantitation ions are used to compute the concentrations of the analytes after they are identified within a specific retention time window by fitting the spectra of the compound of interest to spectra in a user-generated calibration library. The quantitation ion for 1,1-DCE was 61.

Table 4-1. Saturn 2000 MS Operating Conditions[6]

Scan range, amu	26–300
Scan rate, s/scan	0.8 (3 µscans per analytical scan)
Background mass, amu	25
Segment breaks[a]	70/78/150
Segment tune factors[b]	120/70/100/70 (segment time, %)
	25.0/25.0/25.0/25.0 (segment radio frequency [RF], V)
Automatic gain control target	15000–20000
Emission current, µA	15

[a]The segment breaks recommended by Varian for our mass range and compounds of interest divide the mass range into the following four segments: 26–70, 71–78, 79–150, and 151–300 m/z.
[b]The segment tune factor determines the actual ionization time for one segment; segment RF is an RF voltage that is used to hold ions in the trap during the ionization period.

In order to enhance the sensitivity of the method for 1,1-DCE, the standard analytical procedure described above was modified by adjusting the MS parameters as shown in Table 4-2.

Table 4-2. Adjustments Made to Saturn 2000 MS Operating Parameters

	Standard Method	1,1-DCE Method
Scan range, amu	26–300	47–110
Scan time, s/scan	0.8	0.4
Background mass, amu	25	45
Segment RF values	25.0	45.0
AGC prescan storage level	25.0	43.0

4.2.2 Method Detection Limits

The MDL for 1,1-DCE had been determined with our standard autoGC/MS analytical method in earlier experiments to be 180 pptv.[3] In more recent unpublished experiments, the MDL was determined to be 100 pptv. For the current task, MDLs for 1,1-DCE were determined using the autoGC/MS system and the optimized MS method. To determine the MDL, a canister standard of 1,1-DCE at 23 pptv in a humidified air matrix was analyzed seven times on each of the XonTech 930 multisorbent traps. The MDLs were calculated by using the following formula which is defined in the *Federal Register*:

$$\mathrm{MDL} = t_{(n-1,\ 1-\alpha\ =\ 0.99)}S$$

where S is the standard deviation (SD) of replicate analysis and t is the Student's t-value appropriate to a 99% confidence level and a SD estimate with $n-1$ degrees of freedom ($t = 3.143$). The MDLs were determined to be 7 and 5 pptv for traps 1 and 2, respectively. The results of the MDL experiment are listed in Table 4-3. As defined in Method TO-15, section 10.7.5, the quantitation limits (3 × MDL) for this method are 21 and 15 pptv for traps 1 and 2, respectively. For the discussion that follows, a mean MDL of 6 pptv and a mean quantitation limit of 18 pptv will be used.

Table 4-3. Results of MDL Experiment—23 pptv 1,1-Dichloroethene Standard

	Trap 1	Trap 2
	23	23
	27	23
	28	22
	26	22
	24	22
	23	19
	23	20
Mean (n=7)	25	22
SD	2.1	1.5
MDL (pptv)	7	5

4.2.3 Linearity of Response

Linearity of response on our autoGC/MS system using our standard MS method has been documented for 1,1-DCE over a range of 0.5 to 40 ppbv. For this work, canister standards of 1,1-DCE at nominal concentrations of 10, 25, 100, and 200 pptv were prepared and analyzed using the autoGC/MS system. A linear system response to the standards in the range of 10 to 200 pptv was demonstrated. Graphs of the trap 1 and trap 2 data with linear regression results are shown in Figures 4-1 and 4-2.

4.3 Cleanliness Issues

To verify the cleanliness of our analytical system and canisters, multiple analyses of various samples were performed using the optimized method for 1,1-DCE. Analyses of helium blank samples collected by placing the preconcentrator in helium blank mode, analyses of humidified scientific-grade air (HSA) in canisters, and analyses of a nominal 10-ppbv Photochemical Assessment Monitoring Stations (PAMS)/terpenes canister standard all resulted in either nondetection of 1,1-DCE or detection of 1,1-DCE below the quantitation limit of 18 pptv. Additionally, ambient air samples were analyzed from the manifold in the mobile laboratory using the modified method for enhanced detection of 1,1-DCE. Concentrations of 1,1-DCE ranged from not detected to approximately 20 pptv (which is just above the quantitation limit) in these samples.

In the middle of the study, the trap 2 results for pptv concentrations of 1,1-DCE began to exhibit a positive bias. In an effort to correct the problem of divergence of trap response, a new set of multisorbent traps was installed on the Model 930 concentrator. Similar results were observed for the new set of traps, and we are unable to explain the bias in the trap 2 results. For this reason, we have chosen to report only the trap 1 analytical results for the low-pptv concentrations of 1,1-DCE that were determined using the optimized MS method.

4.4 Informal Storage Stability Studies

An informal storage stability study was conducted for canister samples of HSA containing 10, 25, 100, and 200 pptv of 1,1-DCE. The concentration of 1,1-DCE in 10 canisters from

various vendors compared well from the beginning of the two-month study until the end, but the 1,1-DCE concentration in three canisters from one vendor had decreased substantially by the end of the study.

Figure 4-1. Linearity plot for 1,1-Dichloroethene, Trap 1

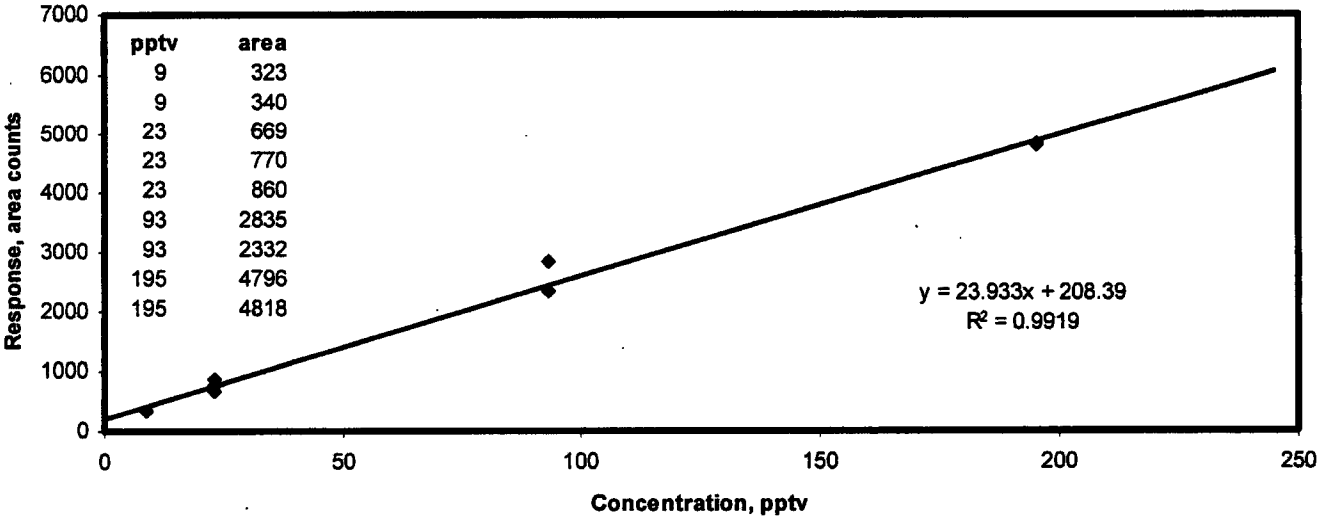
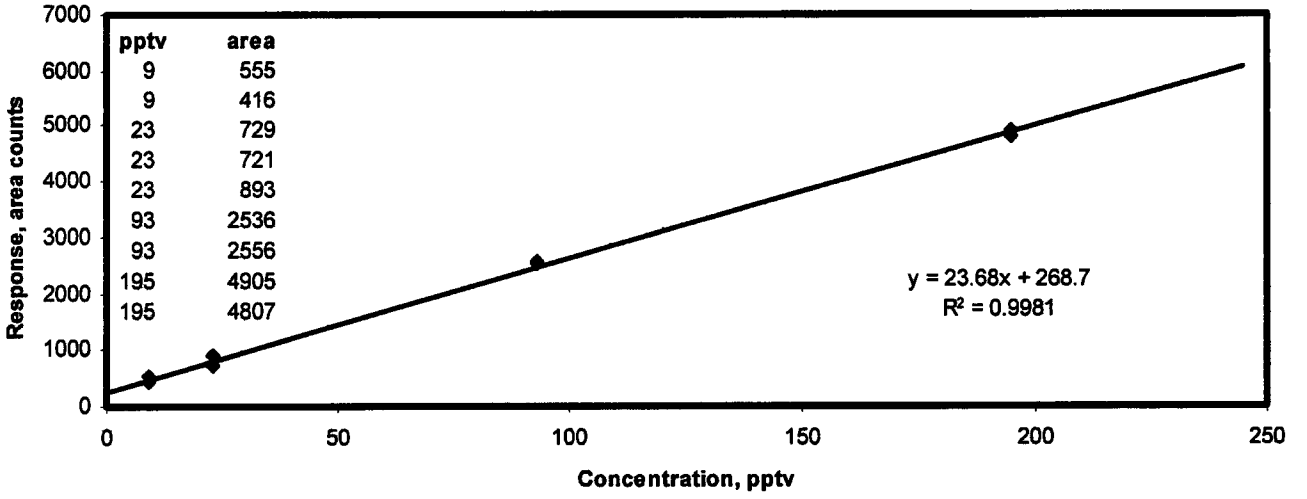


Figure 4-2. Linearity plot for 1,1-Dichloroethene, Trap 2



Chapter 5
Contract Laboratory Experiment

5.1 Experimental Design

An experiment was designed to assess the capabilities of three contract laboratories that provide Method TO-15 type analyses for pptv concentrations of 1,1-DCE in air samples, but *without their knowledge*. The rationale for conducting the experiment in this manner was to ensure that the samples and analytical results would be treated the same as any other samples that the laboratories might receive. The experiment would then result in "real-world" data suitable for use in determining the performance criteria that are necessary to produce valid measurements for low-pptv concentrations of target VOCs. A second criterion for the experiment was that it be conducted with the expenditure of a reasonable amount of money for sample analyses. With these two criteria in mind, an experimental plan was developed that required the preparation of four sets of samples to validate the performance of the laboratories with respect to (1) the cleanliness of their canisters, (2) their ability to determine 1,1-DCE at 20–40 pptv in a humid ambient air matrix, (3) their ability to determine 1,1-DCE at 50–80 pptv in the presence of a mixture of 14 chlorinated VOCs in a humid ambient air matrix, and (4) their ability to determine 1,1-DCE at low-ppbv levels in the presence of ppbv levels of 60 hydrocarbons in a synthetic air matrix.

5.2 Experimental Method

In preparation for the experiment, the three contract laboratories were contacted, and purchase orders were prepared for rental of three canisters from each laboratory as well as for the analysis of nine canister samples by each laboratory.

In our laboratory, calibration standards for the autoGC/MS system were prepared in canisters and included a 10-ppbv PAMS/terpenes standard, a 10-ppbv 1,1-DCE standard, a 500-pptv TO-14 standard, and a 100-pptv 1,1-DCE standard. Additionally, 18 canisters were cleaned for use in the experiment. The canister filling pump apparatus, which consisted of a Metal Bellows Corporation (Sharon, MA) Model MB-151 pump, a 500 sccm Tylan mass flow controller (Millipore Corporation, Bedford, MA), .25-inch stainless steel tube fittings, and .25-inch FEP tubing, was purged thoroughly with HSA from the dynamic dilution manifold in preparation for the experiments. Canisters of HSA were prepared and analyzed to verify the cleanliness of the manifold and canister filling pump system.

By using a glass manifold in our mobile laboratory that is set up to continually pull in ambient outdoor air,[7] we were able to spike the ambient air by using high-pressure cylinders of either 1,1-DCE or a mixture of 14 chlorinated VOCs that contained 1,1-DCE. The flow rates of the spike gases from the cylinders were controlled with mass flow controllers. The spiked ambient air in the manifold was monitored using the autoGC/MS system to determine the appropriate mass flow controller settings that were needed to achieve the target 1,1-DCE concentrations for the experiment.

5.3 Canister Sample Preparation

The canister sample preparation schedule was carefully planned to prevent the possibility of carryover of VOCs in the analytical systems and the canister filling pump system from one experiment to the following experiments. This was accomplished by pressurizing the sets of canisters with air samples containing the lowest concentrations of VOCs at the start of the sample preparation portion of the experiment and by pressurizing the set of canisters with the greatest concentration of VOCS at the end of the sample preparation portion of the experiment. The canister sample preparation procedures for each of the four samples are described below.

5.3.1 Test of Canister Cleanliness

Initially, the nine canisters rented from the three contract laboratories were pressurized simultaneously with HSA from the dynamic dilution manifold. The humidity and temperature of the air in the manifold were 35% RH and 25.3 °C during the filling cycle. The canisters were pressurized to 23 psig over a 6-h period. The GC-FID system with an electron capture detector (ECD) was used to monitor the dynamic dilution manifold as the canisters were being filled.

5.3.2 Test of Ability to Determine 1,1-DCE at 20–40 pptv in a Humid Ambient Air Matrix

The filling pump apparatus was moved to the mobile laboratory, and nine canisters were pressurized simultaneously with ambient air spiked with 1,1-DCE from the manifold in the mobile laboratory. The humidity and temperature of the air in the manifold were 60% RH and 25.3 °C during the filling cycle. The canisters were pressurized to 17 psig over a 5-h period. The autoGC/MS system was used to analyze the air in the manifold on an hourly basis as the canisters were being pressurized from the manifold. The concentrations of 1,1-DCE in the trap 1 samples collected during the 6-h period bracketing the fill cycle were 32, 30, and 38 pptv, with a mean concentration of 33 pptv.

5.3.3 Test of Ability to Determine 1,1-DCE at 50–80 pptv in the Presence of a Mixture of 14 Chlorinated VOCs in a Humid Ambient Air Matrix

Next, six canisters were pressurized simultaneously with ambient air spiked with a mixture of chlorinated compounds, one of which was 1,1-DCE, from the manifold in the mobile laboratory. The following are the 14 chlorinated compounds in the spike gas mixture:

- Chloroethene
- 1,1-Dichloroethene
- 1,1,2-Trichloro-1,2,2-trifluoroethane
- Trichloromethane
- 1,2-Dichloroethane
- 1,1,1-Trichloroethane
- Carbon tetrachloride
- Trichloroethene
- *cis*-1,3-Dichloropropene
- *trans*-1,3-Dichloropropene
- 1,2-Dibromoethane
- Tetrachloroethene
- Chlorobenzene
- Benzyl chloride

The humidity and temperature of the air in the manifold were 70% RH and 25.0 °C during the canister filling cycle. The six canisters were pressurized to 16 psig over a 3.5-h period. The autoGC/MS system was used to analyze the air in the manifold on an hourly basis as the canisters were being pressurized from the manifold. The concentrations of 1,1-DCE in the trap 1 samples collected during the time period bracketing the fill cycle were 71 and 66 pptv, with a mean concentration of 68 pptv.

5.3.4 Test of Ability to Determine 1,1-DCE at Low-ppbv Levels in the Presence of ppbv Levels of 60 Hydrocarbons in a Synthetic Air Matrix Using a Method TO-14 Analysis

Finally, the filling pump apparatus was returned to the laboratory, and three canisters were pressurized from the dynamic dilution manifold with 10 ppbv of a 60-component PAMS/terpenes mixture. The humidity and temperature of the air in the manifold were 35% RH and 25.5 °C during the canister filling cycle. The three canisters were pressurized to ~1 psig over a 50-min period. Afterwards, these three canisters were vented to 0 psig and then pressurized to 15 psig with the 10-ppb cylinder standard of 1,1-DCE in order to generate a final nominal concentration of 5 ppbv per compound for both the PAMS/terpenes 60-component mixture and 1,1-DCE. The GC-FID/ECD system was used to monitor the contents of the dynamic dilution manifold while the canisters were being pressurized with the PAMS/terpenes mixture.

5.4 Canister Sample Analyses

Following canister sample preparation, all 27 canister samples were analyzed on the autoGC/MS system on both of the multisorbent traps, for a total of 54 analyses. The 15 spiked ambient air canister samples were analyzed using the DCE method, which is the MS scan method that was optimized for detection of 1,1-DCE and is discussed in section 4.2.1. The nine HSA canister samples were analyzed using both the DCE method and the TO-15 method, which is the standard MS scan method used in this laboratory and is also discussed in section 4.2.1. The use of the two analytical methods for the HSA samples allowed the determination of 1,1-DCE at low-pptv levels as well as a determination of canister cleanliness for additional VOCs. HSA in a canister from our laboratory was also analyzed as a laboratory method blank, and the background values of analytes found in the blank analyses were subtracted from the analytical results for the HSA samples that were analyzed in this laboratory. The three samples containing ppbv levels of the PAMS/terpenes mixture and 1,1-DCE were analyzed using the standard TO-15 scan method.

After all of the samples were analyzed, the canisters were assigned code names and dates. The canisters were then shipped by overnight carrier to the three laboratories. A summary description of the canister samples that includes canister number, sample contents, sample preparation and analysis dates, MS method, canister pressure both before and after analysis, sample and date codes, laboratory code, and laboratory analysis date is presented in Table 5-1. The time between the analysis of a canister sample in our laboratory and the analysis of the same canister sample in a contract laboratory ranged from 5 to 25 days.

Table 5-1. Contract Laboratory Experiment Sample Canisters

Canister	Sample	Date Filled	Date Analyzed	Method Trap 1	Method Trap 2	Analysis (psig)	Final (psig)	Sample Code	Date Code	Contract Lab Code #	Contract Lab Analysis Date
A-701	Ambient Air + 1,1-Dichloroethene	9/17/02	9/18/02	DCE	DCE	17.0	14.5	House A-2	9-17	1	10-2-02
785	Ambient Air + 1,1-Dichloroethene	9/17/02	9/18/02	DCE	DCE	17.0	15.0	House A-5	9-20	1	10-2-02
GA-B	Ambient Air + 1,1-Dichloroethene	9/17/02	9/18/02	DCE	DCE	17.0	15.0	House A-7	9-24	1	10-2-02
120	Ambient Air + 1,1-Dichloroethene	9/17/02	9/18/02	DCE	DCE	17.0	15.0	House B-2	9-17	2	9-30-02
01578	Ambient Air + 1,1-Dichloroethene	9/17/02	9/18/02	DCE	DCE	16.5	15.0	House B-5	9-20	2	9-30-02
MTC-22	Ambient Air + 1,1-Dichloroethene	9/17/02	9/19/02	DCE	DCE	17.0	15.0	House B-7	9-24	2	9-30-02
208	Ambient Air + 1,1-Dichloroethene	9/17/02	9/19/02	DCE	DCE	17.0	15.0	House C-2	9-17	3	10-9-02
013	Ambient Air + 1,1-Dichloroethene	9/17/02	9/19/02	DCE	DCE	17.0	15.0	House C-5	9-20	3	10-9-02
454	Ambient Air + 1,1-Dichloroethene	9/17/02	9/19/02	DCE	DCE	17.0	15.0	House C-7	9-24	3	10-14-02
N-3	Ambient Air + Chlorinated Cmpds	9/17/02	9/23/02	DCE	DCE	16.0	14.0	House A-1	9-16	1	10-2-02
726	Ambient Air + Chlorinated Cmpds	9/17/02	9/23/02	DCE	DCE	16.0	14.0	House A-4	9-19	1	10-2-02
096	Ambient Air + Chlorinated Cmpds	9/17/02	9/23/02	DCE	DCE	16.0	14.0	House B-1	9-16	2	9-30-02
727	Ambient Air + Chlorinated Cmpds	9/17/02	9/23/02	DCE	DCE	16.0	14.0	House B-4	9-19	2	9-30-02
9682 B	Ambient Air + Chlorinated Cmpds	9/17/02	9/23/02	DCE	DCE	16.0	14.0	House C-1	9-16	3	10-9-02
9677 B	Ambient Air + Chlorinated Cmpds	9/17/02	9/23/02	DCE	DCE	16.0	14.0	House C-4	9-19	3	10-9-02
5226	Humidified Scientific Air (HSA)	9/16/02	9/24/02	DCE	TO-15	22.0	17.5	House A-3	9-18	1	10-2-02
5962	Humidified Scientific Air (HSA)	9/16/02	9/24/02	DCE	TO-15	23.0	21.0	House A-6	9-23	1	10-2-02
1299	Humidified Scientific Air (HSA)	9/16/02	9/24/02	DCE	TO-15	22.5	20.5	House A-8	9-25	1	10-2-02
063240	Humidified Scientific Air (HSA)	9/16/02	9/24/02	DCE	TO-15	22.0	20.0	House B-3	9-18	2	9-30-02
0102	Humidified Scientific Air (HSA)	9/16/02	9/24/02	DCE	TO-15	22.5	20.5	House B-6	9-23	2	9-30-02
02303	Humidified Scientific Air (HSA)	9/16/02	9/24/02	DCE	TO-15	18.0	16.0	House B-8	9-25	2	9-30-02
JMTC 034	Humidified Scientific Air (HSA)	9/16/02	9/25/02	DCE	TO-15	22.0	20.0	House C-3	9-18	3	10-9-02
JMTC 027	Humidified Scientific Air (HSA)	9/16/02	9/25/02	DCE	TO-15	23.0	21.0	House C-6	9-23	3	10-14-02
JMTC 035	Humidified Scientific Air (HSA)	9/16/02	9/25/02	DCE	TO-15	22.5	21.0	House C-8	9-25	3	10-14-02
801	PAMS + Terpenes + 1,1-DCE	9/18/02	9/25/02	TO-15	TO-15	15.0	12.5	Garage A	9-26	1	10-4-02
465	PAMS + Terpenes + 1,1-DCE	9/18/02	9/25/02	TO-15	TO-15	15.0	13.0	Garage B	9-26	2	9-30-02
321	PAMS + Terpenes + 1,1-DCE	9/18/02	9/25/02	TO-15	TO-15	15.0	10.0	Garage C	9-26	3	10-4-02

5.5 Analytical Results

The MDLs for 1,1-DCE reported by the contract laboratories for the low-level TO-15 SIM methods used here ranged from 0.5 to 3 pptv. Since the three contract laboratories supplied analytical results in different formats, we chose to present the results as integer values; therefore, some results were rounded to the nearest integer value. Statistical treatments of the data were performed on the integer values that are presented in the tables.

5.5.1 Test of Canister Cleanliness

Table 5-2 summarizes the results of the analyses of the HSA samples by all four laboratories. The results obtained using our standard TO-15 MS scan method for 35 VOCs are included along with the Method TO-15 SIM results for the 10–14 compounds reported by the contract laboratories. (The low-level TO-15 SIM compound list varies slightly among the three contract laboratories.) As in Table 5-1, the contract laboratories are designated as 1, 2, and 3; our laboratory is designated MT. Reporting limits (RLs), PQLs, and/or MDLs also are included where applicable in Table 5-2: Laboratories 1 and 2 provided RLs with their analytical results and laboratory 3 provided both MDLs and PQLs, with the results that fell between the two values flagged as semi-quantitative.

Overall, the canisters from the contract laboratories were found to be clean. 1,1-DCE was not detected in any of the canisters by any of the four laboratories. For the TO-15 SIM results for additional VOCs, laboratory 1 reported dichloromethane, benzene, and trichloroethene above the RL in each of the three samples; laboratory 2 reported no analytes above the RL in any of the three samples; and laboratory 3 reported only chloroethane above the PQL in one of the three samples. Our TO-15 scan results showed toluene, *m,p*-xylene, and 1,2,4-trimethylbenzene in laboratory 1 canister samples; *m,p*-xylene and toluene in laboratory 2 canister samples; and toluene in two of the laboratory 3 canister samples. More specifically, in our analyses toluene was 714 pptv in one of the laboratory 2 canister samples and the remaining VOCs that were detected in the HSA canister samples were less than 165 pptv. The concentration of toluene that was detected in the laboratory 2 canister sample would not have passed the canister cleanliness acceptance criteria for a standard Method TO-14 or TO-15 type of analysis, both of which specify that target compounds be present at less than 0.2 ppbv; however, toluene was not on the low-level TO-15 SIM target list for any of the three contract laboratories.

Table 5-2. Results for Humidified Scientific Air Samples Analyzed by Contract Laboratory Method TO-15 SIM and by a Scan Method (Results in pptv)

#	Compound	MDL	House A-3 TO-15 Scan JMTC-5226 MT	House A-3 TO-15 SIM JMTC-5226 1	House A-6 TO-15 Scan JMTC-5962 MT	House A-6 TO-15 SIM JMTC-5962 1	House A-8 TO-15 Scan JMTC-1299 MT	House A-8 TO-15 SIM JMTC-1299 1	RL 1	House B-3 TO-15 Scan JMTC-063240 MT	House B-3 TO-15 SIM JMTC-063240 2	House B-6 TO-15 Scan JMTC-0102 MT	House B-6 TO-15 SIM JMTC-0102 2	House B-8 TO-15 Scan JMTC-02303 MT	House B-8 TO-15 SIM JMTC-02303 2	RL 2	House C-3 TO-15 Scan JMTC-034 MT	House C-3 TO-15 SIM JMTC-034 3	House C-6 TO-15 Scan JMTC-027 MT	House C-6 TO-15 SIM JMTC-027 3	House C-8 TO-15 Scan JMTC-035 MT	House C-8 TO-15 SIM JMTC-035 3	PQL 3
0	Bromodichloromethane		NA	NA	NA	NA	NA	NA	NA	NA	ND	NA	ND	NA	ND	11	NA	NA	NA	NA	NA	NA	NA
1	1,2-Dichloro-1,1,2,2-tetrafluoroethane	61	ND		ND		ND			ND		ND		ND			ND		ND		ND		
2	Chloroethene	106	ND	ND	ND	ND	ND	ND	10	ND	ND	ND	ND	ND	ND	7.8	ND	2	ND	ND	ND	ND	10
3	Bromomethane	344	ND		ND		ND			ND		ND		ND			ND		ND		ND		
4	Chloroethane			NA		NA		NA	NA		NA		NA		NA	NA		11		6		12	10
5	Trichlorofluoromethane	65	ND		ND		ND			ND		ND		ND			ND		ND		ND		
6	1,1-Dichloroethene	100	ND	ND	ND	ND	ND	ND	10	ND	ND	ND	ND	ND	ND	10	ND	ND	ND	ND	ND	ND	10
7	Dichloromethane	722	ND	36	ND	36	ND	44	20	ND	ND	ND	ND	ND	ND	120	ND	NA	ND	NA	ND	NA	NA
8	1,1,2-Trichloro-1,2,2-trifluoroethane	38	ND		ND		ND			ND		ND		ND			ND		ND		ND		
9	1,1-Dichloroethane	41	ND	ND	ND	ND	ND	ND	10	ND	ND	ND	ND	ND	ND	20	ND	ND	ND	ND	ND	ND	10
10a	*cis*-1,2-Dichloroethene	82	ND	ND	ND	ND	ND	ND	10	ND	ND	ND	ND	ND	ND	14	ND	ND	ND	ND	ND	ND	10
10b	*trans*-1,2-Dichloroethene		NA	NA	NA	NA	NA	NA	NA	NA	ND	NA	ND	NA	ND	14	NA	ND	NA	ND	NA	ND	10
11	Trichloromethane	44	ND	ND	ND	ND	ND	ND	10	ND	ND	ND	ND	ND	ND	14	ND	NA	ND	NA	ND	NA	NA
12	1,2-Dichloroethane	60	ND	ND	ND	ND	ND	ND	10	ND	ND	ND	ND	ND	ND	20	ND	ND	ND	ND	ND	ND	20*
13	1,1,1-Trichloroethane	48	ND	ND	ND	ND	ND	ND	10	ND	ND	ND	ND	ND	ND	110	ND	ND	ND	ND	ND	ND	10
14	Benzene	55	ND	36	ND	24	ND	24	20	67	ND	ND	ND	ND	ND	62	ND	NA	ND	NA	ND	NA	NA
15	Carbon tetrachloride	33	ND		ND		ND			ND		ND		ND			ND		ND		ND		
16	1,2-Dichloropropane	93	ND		ND		ND			ND		ND		ND			ND		ND		ND		
17	Trichloroethene	29	ND	25	ND	20	ND	116	10	ND	ND	ND	ND	ND	ND	48	ND	ND	ND	ND	ND	ND	50
18	*cis*-1,3-Dichloropropene	48	ND		ND		ND			ND		ND		ND			ND		ND		ND		
19	*trans*-1,3-Dichloropropene	86	ND		ND		ND			ND		ND		ND			ND		ND		ND		
20	1,1,2-Trichloroethane	41	ND	NA	ND	NA	ND	NA	NA	ND	ND	ND	ND	ND	ND	18	ND	NA	ND	NA	ND	NA	NA
21	Toluene	44	122		ND		106			714		145		ND			ND		48		56		
22	1,2-Dibromoethane	47	ND		ND		ND			ND		ND		ND			ND		ND		ND		
23	Tetrachloroethene	101	ND	12	ND	ND	ND	ND	10	ND	ND	ND	ND	ND	ND	100	ND	31	ND	6	ND	4	50
24	Chlorobenzene	83	ND		ND		ND			ND		ND		ND			ND		ND		ND		
25	Ethylbenzene	57	ND		ND		ND			ND		ND		ND			ND		ND		ND		
26	*m,p*-Xylene	62	66		ND		ND			138		ND		ND			ND		ND		ND		
27	Styrene	93	ND		ND		ND			ND		ND		ND			ND		ND		ND		
28	1,1,2,2-Tetrachloroethane	69	ND		ND		ND			ND		ND		ND			ND		ND		ND		
29	*o*-Xylene	66	ND		ND		ND			ND		ND		ND			ND		ND		ND		
30	1,3,5-Trimethylbenzene	44	ND		ND		ND			ND		ND		ND			ND		ND		ND		
31	1,2,4-Trimethylbenzene	70	ND		162		ND			ND		ND		ND			ND		ND		ND		
32	*m*-Dichlorobenzene	38	ND		ND		ND			ND		ND		ND			ND		ND		ND		
33	*p*-Dichlorobenzene	80	ND		ND		ND			ND		ND		ND			ND		ND		ND		
34	*o*-Dichlorobenzene	58	ND		ND		ND			ND		ND		ND			ND		ND		ND		
35	1,2,4-Trichlorobenzene	62	ND		ND		ND			ND		ND		ND			ND		ND		ND		
36	Hexachlorobutadiene	79	ND		ND		ND			ND		ND		ND			ND		ND		ND		

ND = not detected, or detected amount below either MDL or RL
NA = sample not analyzed for this compound
*For sample # C-3: PQL = 10 pptv

5.5.2 Summary of Results for Samples Spiked with 1,1-DCE

Table 5-3 summarizes the analytical results for 1,1-DCE for all samples analyzed by all four laboratories. For the samples of ambient air spiked with 1,1-DCE, the analytical results for the optimized TO-15 scan method ranged from 27 to 32 pptv with a mean of 30 pptv of 1,1-DCE for the nine samples. For the low-level Method TO-15 SIM analyses, laboratory 1 results were 30 pptv for all three samples; laboratory 2 results ranged from 27 to 29 pptv; and laboratory 3 results ranged from 27 to 29 pptv. The mean of the contract laboratory results for 1,1-DCE for the nine canister samples was 29 pptv, and the mean of the results for 1,1-DCE from all four laboratories (a total of 18 measurements) was 29 pptv. A further statistical treatment of the data is presented in section 5.5.3.

For the samples of ambient air spiked with the chlorinated compound mixture, the analytical results for the optimized TO-15 scan method ranged from 57 to 69 pptv for all six samples with a mean of 62 pptv for 1,1-DCE. For the low-level Method TO-15 SIM analyses, laboratory 1 results were 59 and 60 pptv; laboratory 2 results were 53 and 60 pptv; and laboratory 3 results were 54 and 60 pptv. The mean of the contract laboratory results for 1,1-DCE for the six canister samples was 58 pptv, and the mean of the results for 1,1-DCE from all four laboratories (a total of 12 measurements) was 60 pptv. A further statistical treatment of the data is presented in section 5.5.4.

For the three PAMS/terpenes plus 1,1-DCE samples, the 1,1-DCE analytical results (trap 1 results only) for our standard TO-15 scan method were 5100 and 5200 pptv, and the contract laboratories' 1,1-DCE results were 5600, 6000, and 11700 pptv.

As stated earlier, 1,1-DCE was not detected in any of the HSA canister samples by any of the four laboratories.

5.5.3 Test of Ability to Determine 1,1-DCE at 20–40 pptv in a Humid Ambient Air Matrix

Table 5-4 presents the analytical results for 10–14 VOCs reported by the three contract laboratories for the canister samples of ambient air spiked with 1,1-DCE. As mentioned earlier, the low-level TO-15 SIM compound list varies slightly among the three contract laboratories. Data on VOCs other than 1,1-DCE are not available from our laboratory as our method development and calibration efforts at the low-pptv level were directed solely toward the determination of 1,1-DCE.

The RSD of the measurements was calculated as follows:

$$\text{RSD} = \text{SD/mean} \times 100$$

and is included in the table as a measure of replicate precision for the VOC results reported by each of the contract laboratories. (*Note*: Replicate precision is defined in section 5.10 of Method TO-15 as precision determined from two canisters, whereas duplicate precision is defined in section 5.11 as precision determined from the analysis of two samples taken from the same canister.) The RSD for 1,1-DCE was 0.0, 3.6, and 4.2% for laboratories 1, 2, and 3, respectively. The RSDs for other compounds reported by the contract laboratories were less than 12% for all measurements that were above the RL/PQL.

RSDs were also calculated for the 1,1-DCE results that are summarized in Table 5-3. For the nine canister samples analyzed in our laboratory using the optimized 1,1-DCE scan method, the RSD was 5.7%. The RSD for the combined contract laboratory results for the nine canister samples analyzed using the low-level TO-15 SIM methods was 4.2%. The RSD of the results from all four laboratories (a total of 18 measurements) was 5.5%.

5.5.4 Test of Ability to Determine 1,1-DCE at 50–80 pptv in the Presence of a Mixture of 14 Chlorinated VOCs in a Humid Ambient Air Matrix

Table 5-5 presents the analytical results for 10–14 VOCs reported by the three contract laboratories for the canister samples of ambient air spiked with a mixture of chlorinated compounds. As mentioned earlier, the low-level TO-15 SIM compound list varies slightly among the three contract laboratories. Data on VOCs other than 1,1-DCE are not available from our laboratory as our method development and calibration efforts at the low-pptv level were directed solely toward the determination of 1,1-DCE.

As a measure of replicate precision, the percent difference (%D) was calculated according to the definition in section 11.3.1 of Method TO-15 as follows:

$$\%D = \frac{|x_1 - x_2|}{\bar{x}} \times 100$$

where x_1 is the first measurement value, x_2 is the second measurement value, and $\bar{x}$ is the average of the two values. The %D for 1,1-DCE was 0.0, 12.4, and 10.4% for laboratories 1, 2, and 3, respectively. The %D in the measurements for the other compounds reported by the contract laboratories was less than 25% for all of the replicate measurements except one (45.6% for tetrachloroethene for laboratory 1), and 20 of 23 replicate measurements (including 1,1-DCE) had a %D of <15%.

RSDs were calculated for the 1,1-DCE results that are summarized in Table 5-3. For the six canister samples analyzed using the optimized 1,1-DCE scan method, the RSD was 6.7%. The RSD for the combined contract laboratory results for the six canister samples analyzed using the low-level TO-15 SIM methods was 5.5%. The RSD of the results from all four laboratories (a total of 12 measurements) was 6.8%.

5.5.5 Test of Ability to Determine 1,1-DCE at Low-ppbv Levels in the Presence of ppbv Levels of 60 Hydrocarbons in a Synthetic Air Matrix Using a Method TO-14 Analysis

Table 5-6 summarizes the MS scan results for the PAMS/terpenes plus 1,1-DCE samples for all four laboratories. Since a TO-14 type analysis was requested of the contract laboratories for these three canister samples, most of the 60 hydrocarbons in the mixture were not on the target lists. The 1,1-DCE results were 5.6, 6.0, and 11.7 ppbv for the contract laboratories. The 1,1-DCE results obtained in this laboratory using the standard Method TO-15 scan method ranged from 4.5–5.9 ppbv with a mean of 5.1 ppbv for the eight analyses. Of particular concern is the 11.7-ppbv concentration measured by laboratory 3 for 1,1-DCE, as that measurement is approximately twice the concentration determined by the other three laboratories. Additionally, for all three contract laboratories certain measurements for various compounds (benzene, 6.8 ppbv; toluene, 6.0 ppbv; 4-ethyltoluene, 9.3 ppbv; 1,3,5-trimethylbenzene, 6.3 ppbv; and 1,2,4-trimethylbenzene, 7.3 ppbv) were somewhat higher than those determined by the other laboratories.

Table 5-3. Analytical Results for 1,1-Dichloroethene

#	Canister	Sample	MT (Trap 1) (pptv)	Contract Lab (pptv)	Contract Lab #
1	A-701	Ambient Air + 1,1-Dichloroethene	32	30	1
2	785	Ambient Air + 1,1-Dichloroethene	27	30	1
3	GA-B	Ambient Air + 1,1-Dichloroethene	30	30	1
4	120	Ambient Air + 1,1-Dichloroethene	30	28	2
5	01578	Ambient Air + 1,1-Dichloroethene	32	29	2
6	MTC-22	Ambient Air + 1,1-Dichloroethene	29	27	2
7	208	Ambient Air + 1,1-Dichloroethene	32	27	3
8	013	Ambient Air + 1,1-Dichloroethene	31	29	3
9	454	Ambient Air + 1,1-Dichloroethene	29	29	3
10	N-3	Ambient Air + Chlorinated Cmpds	69	59	1
11	726	Ambient Air + Chlorinated Cmpds	59	59	1
12	096	Ambient Air + Chlorinated Cmpds	60	60	2
13	727	Ambient Air + Chlorinated Cmpds	61	53	2
14	9682 B	Ambient Air + Chlorinated Cmpds	57	60	3
15	9677 B	Ambient Air + Chlorinated Cmpds	62	54	3
16	5226	Humidified Scientific Air (HSA)	ND	ND	1
17	5962	Humidified Scientific Air (HSA)	ND	ND	1
18	1299	Humidified Scientific Air (HSA)	ND	ND	1
19	063240	Humidified Scientific Air (HSA)	ND	ND	2
20	0102	Humidified Scientific Air (HSA)	ND	ND	2
21	02303	Humidified Scientific Air (HSA)	ND	ND	2
22	JMTC 034	Humidified Scientific Air (HSA)	ND	ND	3
23	JMTC 027	Humidified Scientific Air (HSA)	ND	ND	3
24	JMTC 035	Humidified Scientific Air (HSA)	ND	ND	3
25	801	PAMS + Terpenes + 1,1-DCE	5200	5600	1
26	465	PAMS + Terpenes + 1,1-DCE	5100	6000	2
27	321	PAMS + Terpenes + 1,1-DCE	5200	11700	3

ND = not detected, or detected amount below either MDL or RL.
MT = ManTech.

Table 5-4. Contract Laboratory Method TO-15 SIM Analytical Results for Ambient Air Spiked with 1,1-Dichloroethene (Results in pptv)

SAMPLE NAME	House A-2	House A-5	House A-7			House B-2	House B-5	House B-7			House C-2	House C-5	House C-7		
CANISTER	A-701	785	GA-B	RSD	RL	120	01578	MTC-22	RSD	RL	208	013	454	RSD	RL
Compound LAB	1	1	1		1	2	2	2		2	3	3	3		3
Bromodichloromethane	NA	NA	NA		NA	ND	ND	ND		11	NA	NA	NA		NA
Chloroethene	ND	ND	ND		10	ND	ND	ND		7.8	ND	ND	ND		10
Chloroethane	NA	NA	NA		NA	NA	NA	NA		NA	7^	7^	6^	8.7	10
1,1-Dichloroethene	30	30	30	0.0	10	28	29	27	3.6	10	27	29	29	4.2	10
Dichloromethane	184	175	183	2.7	20	120	120	120	0.0	120	NA	NA	NA		NA
1,1-Dichloroethane	ND	ND	ND		10	ND	ND	ND		20	ND	ND	ND		10
cis-1,2-Dichloroethene	10	11	ND		10	ND	ND	ND		14	ND	ND	ND		10
trans-1,2-Dichloroethene	NA	NA	NA		NA	ND	ND	ND		14	ND	ND	ND		10
Trichloromethane	ND	25	17		10	30	30	28	3.9	14	NA	NA	NA		NA
1,2-Dichloroethane	ND	ND	ND		10	ND	ND	ND		20	3^	3^	ND		10*
1,1,1-Trichloroethane	38	36	36	3.1	10	ND	ND	ND		110	27	28	25	5.7	10
Benzene	311	288	297	3.9	20	230	230	230	0.0	62	NA	NA	NA		NA
Trichloroethene	ND	ND	41		10	ND	ND	ND		48	2^	2^	ND		50
1,1,2-Trichloroethane	NA	NA	NA		NA	ND	ND	ND		18	NA	NA	NA		NA
Tetrachloroethene	27	32	34	11.6	10	ND	ND	ND		100	39^	32^	18^	36.0	50

ND = not detected, or detected amount below either MDL or RL.
NA = sample not analyzed for this compound.
^Semi-quantitative sample result value (value between MDL and PQL).
*For sample # C-7, PQL = 20 pptv.

Table 5-5. Contract Laboratory Method TO-15 SIM Analytical Results for Ambient Air Spiked with a Chlorinated Gas Mixture (Results in pptv)

SAMPLE NAME	House A-1	House A-4			House B-1	House B-4			House C-1	House C-4		
CANISTER	N-3	726	%D	RL	096	727	%D	RL	9682-B	9677-B	%D	PQL
Compound LAB	1	1		1	2	2		2	3	3		3
Bromodichloromethane	NA	NA		NA	ND	ND		11	NA	NA		NA
Chloroethene	60	66	9.5	10	64	62	3.2	7.8	59	65	9.7	10
Chloroethane	NA	NA		NA	NA	NA		NA	7^	8^	13.3	10
1,1-Dichloroethene	59	59	0.0	10	60	53	12.4	10	60	54	10.5	10
Dichloromethane	147	161	9.1	20	ND	ND		120	NA	NA		NA
1,1-Dichloroethane	ND	ND		10	ND	ND		20	ND	ND		10
cis-1,2-Dichloroethene	14	16	13.3	10	ND	ND		14	ND	ND		10
trans-1,2-Dichloroethene	NA	NA		NA	ND	ND		14	ND	ND		10
Trichloromethane	31	38	20.3	10	87	91	4.5	14	NA	NA		NA
1,2-Dichloroethane	87	89	2.3	10	70	73	4.2	20	64	61	4.8	10
1,1,1-Trichloroethane	113	117	3.5	10	ND	ND		110	91	87	4.5	10
Benzene	276	263	4.8	20	200	190	5.1	62	NA	NA		NA
Trichloroethene	90	78	14.3	10	58	59	1.7	48	52	48^	8.0	50
1,1,2-Trichloroethane	NA	NA		NA	ND	ND		18	NA	NA		NA
Tetrachloroethene	175	110	45.6	10	ND	ND		100	98	77	24.0	50

ND = not detected, or detected amount below either MDL or RL.
NA = sample not analyzed for this compound.
^Semi-quantitative sample result value (value between MDL and PQL).

Table 5-6. Analytical Results for the PAMS/Terpenes + 1,1-Dichloroethene Mixture (Results in ppbv)

SAMPLE NAME	Garage A	Garage A	Garage A	Garage B	Garage B	Garage B	Garage C	Garage C	Garage C	Garage C	Garage C
CAN	801	801	801	465	465	465	321	321	321	321	321
Compound LAB	MT	MT	1	MT	MT	2	MT	MT	MT	MT	3
1,1-Dichloroethene	5.2	4.8	5.6	5.1	4.9	6.0	5.2	4.5	5.9	5.1	11.7
Benzene	4.7	4.7	4.9	4.8	4.7	4.9	4.5	4.6	5.4	4.9	6.8
Toluene	4.3	4.4	4.6	4.4	4.4	4.6	4.4	4.3	4.8	4.7	6.0
Ethylbenzene	4.2	4.4	5.4	4.4	4.4	4.7	4.1	4.2	4.7	4.6	5.5
m,p-Xylene	8.9	9.0	11	9.3	9.0	15 (total)	9.2	8.9	10.1	9.6	12.1
Styrene	4.1	3.9	4.6	4.4	4.0	4.2	4.1	3.8	4.8	4.1	5.4
o-Xylene	4.4	4.5	5.6	4.7	4.5	see m,p-Xyl	4.5	4.4	5.1	4.9	5.6
4-Ethyltoluene (*p*-)	4.0	3.9	NA	4.1	4.2	9.3	4.1	3.9	4.9	4.5	NA
1,3,5-Trimethylbenzene	4.7	4.4	6.3	5.2	4.3	4.7	5.0	4.3	5.3	4.6	5.3
1,2,4-Trimethylbenzene	4.6	4.6	7.3	5.4	4.4	4.8	5.1	4.4	5.6	4.6	5.9

NA = sample not analyzed for this compound

Chapter 6
References

1. McClenny, W.A., Joachim D. Pleil, Thomas A. Lumpkin, Karen D. Oliver. 1987. Update on canister-based samplers for VOCs. Proceedings of the EPA/APCA Symposium on Measurement of Toxic and Related Air Pollutants, VIP-8, Air & Waste Management Association, Pittsburgh, PA, pp. 253–258.

2. Lonneman, W.A., 2002. Personal communication.

3. Oliver, Karen D. and Joachim D. Pleil. 1985. Automated cryogenic sampling and gas chromatographic analysis of ambient vapor-phase organic compounds: Procedures and comparison tests. TN-4120-85-02 under EPA Contract No. 68-02-4035, Northrop Services, Inc. - Environmental Sciences, P.O. Box 12313, Research Triangle Park, NC 27709.

4. Lonneman, W.A. 1998. Comparison of hydrocarbon composition in Los Angeles for the year 1968 and 1997. Proceedings of the EPA/A&WMA Symposium on Measurement of Toxic and Related Air Pollutants, VIP-85, Air & Waste Management Association, Pittsburgh, PA, pp. 356–365.

5. Oliver, Karen D., Jeffrey R. Adams, E. Hunter Daughtrey, Jr., William A. McClenny, Matthias J. Yoong, Michael A. Pardee, Elizabeth B. Almasi, Norman A. Kirshen. 1996. Technique for monitoring toxic VOCs in air: Sorbent preconcentration, closed-cycle cooler cryofocusing, and GC-MS analysis. *Environ. Sci. Technol.* 30:1939-1945.

6. Saturn GC/MS Reference Manual. 1995. P/N 03-914354-40:3. Walnut Creek, CA: Varian Associates, Inc.

7. McClenny, W.A., Karen D. Oliver, Henry H. Jacumin, Jr., E. Hunter Daughtrey, Jr. 2002. Ambient level volatile organic compound (VOC) monitoring using solid adsorbents—Recent US EPA studies. *J. Environ. Monit.* 4:695–705.

Appendix B
Example Guidance Provided by the Colorado Department of Public Health and Environment (CDPHE) from "Guidance for Analysis of Indoor Air Samples—April 2000"

The CDPHE guidance addresses the analysis of indoor air samples from specially treated canisters by providing a set of technical requirements that place the analytical system under control and allow low-pptv detection of VOCs. These requirements were used by at least one of the laboratories in the laboratory comparison study mentioned in the main text with successful results at monitoring levels of 30–60 pptv. Other sets of technical requirements were also used to achieve these results in the laboratory comparison study, including the technical requirements used by ManTech Environmental Technology, Inc. (the NERL on-site contractor).

B.1 Tuning Requirements for GC/MS-SIM Instruments

CDPHE requires the use of the tuning compound perfluorotributylamine (PFTBA) and tuning algorithms to meet the following conditions: (1) The operator must confirm that the 69/70, 219/220, and 502/503 m/e ion ratios occur at the proper ratios of 1% (± 50%), 5% (±25%), and 10% (±10%), respectively; (2) the peak width at half height for the 502, 219, and 69 PFTBA m/e ions must be 0.5 amu ±0.2 amu; and (3) the operator must confirm the correct mass assignment of these m/e ions to a tolerance of 0.1 amu (e.g., 69.0 amu ±0.1 amu). Also, the operator must verify that the tuning is stable at a minimum of once per operating day to ensure correct mass axis alignment and eliminate data accumulated with contaminated ion sources.

B.2 Data Acquisition Requirements for GC/MS-SIM Instruments

CDPHE requires that GC/MS instruments operated in the SIM mode to meet Compendium Methods to acquire data with 1 amu of resolution and meet the following conditions: (1) the operator must demonstrate compliance with the tuning requirements; (2) the operator must confirm that the software method used to collect calibrant and sample data be set to the high-resolution option (1 amu); (3) the ion dwell times must be optimized to obtain a minimum of 10 scans per peak; and (4) the electron multiplier (EM) voltages must be set to meet the detection limits of the project (conveniently accomplished by setting EM voltages at +300 volts relative to the tune voltage).

B.3 Ion Selection for GC/MS-SIM and GC/MS-Scan

CDPHE has provided a table of characteristic ions for four target compounds. The ions in Table B-1 are used to determine target compound concentrations by GC/MS-Scan and GC/MS-SIM methods.

B.4 Summary of Technical Requirements from CDPHE for Analysis of Indoor Air Samples

CDPHE has provided a summary table of minimal acceptable requirements for analysis of indoor air samples, which is presented in Table B-2.

B.5 Contact Information

CDPHE has agreed to provide the following contact listings so that interested readers can contact them for further information:

Colorado Department of Public Health and Environment
Hazardous Materials and Waste Management Division
Technical Assistance
4300 Cherry Creek Drive South
Denver, CO 80246-1530

Telephone: (303) 692-2000
Toll-free: (800) 886-7689
Fax: (303) 759-5355
Website: http://www.cdphe.state.co.us/

Table B-1. Characteristic Ions for Four Target Compounds

Contaminant	Compendium Characteristic Ions[1]	CDPHE Preferred GC/MS-Scan Equivalent Method Characteristic Ions[2]	GC/MS-SIM Suggested Ions
1,1-DCE	61[3], 96	96, 61, 63	96, 98[5]
1,2-DCA	62, 64	62, 98	62, 98[5] or 62, 64
CH_2Cl_2	49[3], 84[4], 86	84, 86, 49	84, 86
TCE	130, 95[4]	95, 130, 132	130, 132

(1) EPA Air Compendium Methods TO-14, TO-14a, and TO-15. Primary (quantitation ion) listed first.
(2) EPA method(s) 8260B (SW-846), 624 (Clean Water), and 524 (Drinking Water). Primary ion listed first.
(3) Interference detected on the primary (quantitation) ion, evaluation of 3 projects. Data from two laboratories using GC/MS-Scan and GC/MS-SIM.
(4) Interference detected on the secondary (confirming) ion, evaluation of 3 projects. Data from two laboratories using GC/MS-Scan and GC/MS-SIM.
(5) The selection of the 98 ion reflects the prominence of this ion for this compound, and observed interferences.

Table B-2. Minimum Acceptable Requirement for Analysis of Indoor Air Samples

Activity	Specifications	Documentation Needed
GC/MS-SIM Tuning	Autotune or equivalent. Acceptable isotopic ratios (1, 5, 10%) Peak width at half height (0.5 amu +/- 0.2) Correct mass assignment (+/- 0.1 amu)	Printout of tune report
GC/MS-SIM Data Acquisition	Meet tune specifications. Optimize ion dwell time.	Printout of instrument method 10 scans/peak minimum Printout of extracted ion chromatogram.
	Set electron multiplier voltage to achieve required detection limits.	Data quality objectives
	Collect calibrant and sample analysis data with the high-resolution option (1 amu).	Printout of instrument method Raw sample data
Ion Selection		**Reference**
GC/MS-SIM	Select primary ions from 8260B tabular data, or at least two ions, justified from library spectra, that meet data quality objectives. (Free from interferences)	Method 8260B, library spectra
	Consecutively evaluate ion selection. Adjust as necessary.	Library spectra, raw sample data
GC/MS-SCAN	Select primary ions from 8260B tabular data, or at least two ions, justified from library spectra that meet data quality objectives. (Free from interferences)	Method 8260B, library spectra
	Consecutively evaluate ion selection. Adjust as necessary.	Library spectra, raw sample data

Continued on following page

Table B-2. Continued

GC/MS-SIM Reporting Requirements

Confirmed positive detections: (REPORT: Concentration, qualify quantitative estimates with a "J")

- Ion relative retention time (RRT) tracks that of standards ***(+/- 0.10 RRT)***
- Characteristic ion abundance ratio tracks ratio of standards (+/- 25 %)
- Characteristic ions maximize within +/- one scan

Unconfirmed detections: (REPORT: Detected not confirmed, specify reason. Qualify quantitative estimates with a "J")

- Ion relative retention time tracks that of standards ***(+/- 0.10 RRT)***
- Characteristic ion abundance ratio fails to track ratio of standards (+/- 25 %)
- Characteristic ions do not maximize within +/- one scan

U.S. Department
of Transportation
**Research and
Special Programs
Administration**

400 Seventh St., S.W.
Washington, D.C. 20590

FEB 13 2003

Mr. Henry L. Longest, II
Acting Assistant Administrator
U. S. Environmental Protection Agency
1300 Pennsylvania Ave, NW (8101R)
Washington, DC 20460

Ref No.: 02-0093

Dear Mr. Longest :

This is in response to your January 29, 2002 letter regarding the applicability of the Hazardous Materials Regulations (HMR; 49 CFR Parts 171-180) to certain environmental samples. Specifically, you requested confirmation that environmental samples which are preserved at the Environmental Protection Agency (EPA) prescribed guidance concentrations, even when reasonably over-preserved, are not corrosive materials subject to the HMR.

The answer is yes. According to your letter and test results submitted, four preservatives (three acids and one base: Nitric acid; Sulfuric acid; Hydrochloric acid; and Sodium Hydroxide) were each tested in an aqueous solution. The environmental samples were prepared by adding a preservative to distilled water. Preserved samples were tested for corrosivity in accordance with 49 CFR §173.137.

Based on the test results, it is the opinion of this office that the environmental samples containing the following "upper limit" concentrations: 0.28 weight percent Nitric acid, 0.38 weight percent Sulfuric acid, 0.15 weight percent Hydrochloric acid and 0.20 weight percent Sodium hydroxide, do not meet the definition of corrosive material in §173.136, and, therefore, are not subject to the HMR.

I hope this information is helpful. Please contact us if you require additional assistance.

Sincerely,

Edward T. Mazzullo
Director, Office of Hazardous
Materials Standards

SOP No. HW-2

Evaluation of Metals Data for the Contract Laboratory Program(CLP)

based on

SOW - ILM05.3

(SOP Revision 13)

United States Environmental Protection Agency
Region 2

Date: September 2005

PREPARED BY: _____ DATE: _____
Hanif Sheikh, Quality Assurance Chemist
Hazardous Waste Support Section

APPROVED BY: _____ DATE: _____
Linda Mauel, Chief
Hazardous Waste Support Section

APPROVED BY: _____ DATE: _____
Robert Runyon, Chief
Hazardous Waste Support Branch

Table of Contents

1.0 Scope

1.1 This Standard Operating Procedure (SOP) applies to the evaluation of Routine Analytical Services (RAS) inorganic data generated in accordance with the EPA Contract Laboratory Program (CLP) protocols.

1.2 This Region 2 inorganic data validation SOP is used to determine the usability of analytical data generated from water and soil/sediment samples collected from Superfund sites in EPA Region 2.

1.3 Data should be generated and validated in accordance with the site specific Project Quality Objectives (PQOs) developed prior to the sample collection event. This SOP can be customized to validate the data according to the site specific PQOs. If the site specific DQOs are not available, this SOP must be used in its entirety.

1.4 This SOP is based, for the most part, upon analytical and quality assurance requirements specified in the Statement of Work SOW-ILM05.3, as well as in the final (October 2004) of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review. The SOP Checklist, Appendix A.1, provides guidance in conducting the data validation. The result of the use of this SOP is a **Total Review** of the data: **Technical plus Contract - Compliance Review.**

2.0 Contract Compliance Review

This type of review is the first step in data validation which is carried out to ensure that the CLP laboratory has analyzed the environmental samples in accordance with the Statement of Work (SOW), and provided a data package which is both complete and compliant. This means that laboratory's procedures were performed exactly as specified in the CLP Statement of Works (SOW) and the data package contains all the deliverables including the information required under the contract.

2.1 Completeness

The data validator must check the entire data package to ensure that all deliverables required under the CLP contract are present and legible. In addition, copies of the Contract Compliance Screening (CCS) report, re-submittal from the laboratory, and Regional documentation should also be present in the data package. In Region 2, the data package completeness check is currently performed by the Regional Sample Control Coordinator (RSCC) for each Sample Delivery Group (SDG). The data package is not released to the data validator until all the required deliverables are received

from the laboratory.

2.2 **Compliance**

The data validator must check to ensure that all steps from sample receipt through sample preparation, analysis, data calculation and reporting are documented, and the information/data required under the contract is present in the appropriate reporting Forms and laboratory logs.

2.3 **Contract Compliance Screening (CCS)**

This screening step essentially checks the data package for the Completeness and Compliance requirements, and is performed by the Sample Management Office (SMO) currently operated by Computer Sciences Corporation (CSC), an EPA contractor. The CCS Report outlines the incomplete and non-compliant items as "Defects" in the data package, and is sent to the laboratory which is required to provide additional or missing information/data required under the contract. The CCS Report for each SDG is transmitted electronically by the SMO to the Regional office. The CCS Report is intended to aid the data validator in locating any problems, both corrected and uncorrected. The incorrect original deliverable(s)of the data package must be replaced by the re-submittal(s)received from the laboratory in response to the CCS Report. The data validation should, however, be carried out even if the CCS Report is not available.

Web-based CCS is available for CLP laboratories to check their data prior to its delivery to EPA.

3.0 **Technical Review**

Technical review of the RAS data is carried out on the complete and compliant data to ensure its **validity** (i.e., data is of known quality and scientifically valid) and **usability** (i.e., data set is sufficiently complete and of sufficient quality to support a decision or an action described in the specific objectives of a data collection activity). The technical review process provides information on analytical limitations of data, if any, based on specific Quality Assurance/Quality Control (QA/QC) criteria. This is accomplished by performing an in-depth review of both the field deliverables which document the field sampling activities, and the laboratory analytical data deliverables which document the laboratory activities carried out to generate the reported data. Essentially, the validator shall first ensure that the data package is complete and compliant. The validator shall then evaluate data/information on all these deliverables (Final data sheets, Forms for QC analyses Chain-of-Custody/Traffic Report Forms, raw data, etc.) against the QA/QC acceptance criteria specified in the SOP "Checklist" (Appendix A.1). The validator must answer each question in the

" Checklist" and take an appropriate action as required under "Action" to qualify the data. As a result of the technical review, the data validator may qualify some of the data as **rejected** or as **estimated.** The data validator shall write a **Data Review Narrative** documenting the qualified data and the reason(s) for the qualification.

3.1 If the **raw data** necessary to support the reported results are not provided, the data validation must not be performed. The laboratory must be contacted to obtain missing raw data.

3.2 If batch quality control analyses are performed on samples other than **site specific samples**, data must not be validated or at best be considered as estimated. The data user must be notified of this action.

3.3 **QA/QC Acceptance Criteria**
In order that reviews be consistent among reviewers, QA/QC protocol (stated in Appendix A.1) should be strictly adhered to. If a lab provides more than one set of QC analyses or more than one particular QC analysis for an SDG, the validator shall use the worst QC analysis to evaluate the SDG data. Professional judgement should only be used in the rare instances not addressed in the "Checklist".

3.4 **Data Validation Flags**
Three types of data validation flags (J, R & U) are used in Region 2 to qualify the data.

3.4.1 **Flag "R" indicates Rejected Data**
Sample results determined to be unacceptable must preferably be lined over and flagged " R" with a red pencil only on the Inorganic Analysis Data Sheets (CLP Form I's). Data rejected on the basis of an unacceptable QC analysis should be excluded from further review or consideration. Data are rejected when associated QC analysis results exceed the expanded control limits of the QC criteria. The rejected data are known to contain significant errors based on documented information. The data user **must not** use the rejected data to make environmental decisions.

3.4.2 **Flag "J" indicates Estimated Data**
Sample results determined to be estimated must be flagged "J" with a red pencil only on the CLP Form I's. Data are flagged (J) when a QC analysis falls outside the primary acceptance limits. The qualified "J" data are not excluded from further review or consideration. However, only one flag (J) is applied to a sample result even though several associated QC analyses may fail. The "J" data may be biased high or low.

3.4.3 **Flg "U" indicates Non-Detects**
Sample results ≥ MDL associated with a contaminated blank are flagged "U" with a red pencil only on Form I's.

4.0 **Contractual Qualifiers**
The CLP laboratory applies contractual qualifiers on all Form I'S and the QC Forms when QC analyses are outside the control limits. These qualifiers are not applied on the Lotus or XLS spreadsheets with the exception of U and J. The contractual qualifiers and their meanings are as follows:

N : This qualifier indicates the lack of accuracy in the reported result, and is applied when matrix spiked sample recovery is outside the control limits.

E : This qualifier indicates the presence of interference, and is applied when the ICP serial dilution analysis is outside the control limits.

* : This qualifier indicates the lack of precision, and is applied to sample results on Form I's and Form VI when the Lab Duplicate analysis is outside the control limits.

U : This is a concentration qualifier that laboratory applies to a non-detected result which is essentially less than the Method Detection Limit(MDL). A non-detected result of an analysis` is indicated by the Contract Required Quantitation Limit (CRQL) of that analyze suffixed with "U".

J : This is a concentration qualifier that the laboratory applies to a positive result below the CRQL(i.e.,≥MDL but <CRQL).

NOTE: The laboratory qualifiers are crossed out and replaced with the appropriate data validation qualifiers (J, R or U) by the data validator.

4.0 **Rounding Rule**
The data reviewer must follow the standard practice to round off percent recoveries on the QC reporting forms.

5.0 **Data Review Narrative (Appendix A.2)**
The data review narrative should be written using the format of Appendix A.2. The narrative should indicate the QC analyses outside the acceptance limits and the actions taken to qualify the associated data. The narrative should be

prepared on a Personal Computer or a typewriter. If hand-written, under no circumstances should a pencil be used to write the narrative. The Data Review Narrative should be written in four (4) Sections: (i)Data Case Description, (ii)Complete SDG File (CSF) Audit Section, (iii) Technical Review Section, and (iv) Contract-Problems/Non-Compliance Section.

5.1 **Data Case Description Section**
The data validator must briefly describe the data case in this Section, outlining important information such as the number of samples, their matrix, sampling date(s), analysis (TAL metals, mercury or cyanide), samples used for QC analyses, Field Blank(s), Field Duplicates, etc.

5.2 **Complete SDG File (CSF) Audit Section**
The data validator must perform an audit on each SDG in the data package to ensure that all SDG-specific documents (sampling, samples shipping and receiving, telephone contact logs, etc.) are present in the data case. The audit shall also discover any discrepancy in the deliverables. In Region 2, this audit is currently performed by the ESAT data validator and its findings reported under "Comments" on a CSF inventory checklist. The validator informs the CLP Project Officer (PO) of the missing or additional information/deliverable required for data validation. The PO then contacts the lab for the desired deliverable/information. The findings of the CSF audit are reported in the CSF Section of the Data Review Narrative (Appendix A.2).

5.3 **Technical Review Section**
The data validator shall report in this Section only the rejected (R) and estimated data (J) and the data rendered non-detects (U) as a result of technical review. It is imperative that the data reviewer highlights (i) QC analysis criteria applied to reject (R) or flag (J, U) the data, (ii) Samples rejected (R) or flagged (J, U), and (iii) the QC analysis out of control limits. The rest of the data that are not qualified (rejected or estimated) are not reported in this Section, and should be considered **fully useable.**

5.4 **Contract-Problems/Non-Compliance Section**
All the CLP non-compliant items detected during data review must be reported in this Section.

6.0 **Computer-Aided Data Review and Evaluation (CADRE)**
CADRE is a computer program that performs semi-automated Quality Assurance (QA) and Quality Control (QC) checks of results from the chemical analysis of soil and water samples according to the CLP protocols. After the CADRE data

qualification is complete, a Lotus 1,2,3 spreadsheet or an XLS spreadsheet with data validation qualifiers (R,J,U) is generated for each SDG. Currently, Sample Management Office (SMO) performs this task using Data Assessment Tool (DAT), a software-driven process, and forwards to the Regions the customized electronic spreadsheets (Lotus 1,2,3 or XLS spreadsheet) and QC reports via the DART (Data Assessment Rapid Transmittal) system. Manual data validation is performed in conjunction with electronic data validation which can only be done by a trained and experienced data validator. The manual data review complements CADRE's findings to complete an assessment of data quality in a shorter time than by a solely manual process. The data validator must review the XLS or Lotus 1,2,3 spreadsheet against Form I's to ensure that the same results on Form I's and the Spreadsheet are qualified with the same data validation qualifiers. The spreadsheet for each SDG is provided with the Data Review Narrative.

7.0 Performance Evaluation Sample(PES)Based Data Validation Strategy

7.1 Scope and Summary

This strategy offers the use of Performance Evaluation Samples (PES) in the data validation process as a means of ensuring the quality of the CLP data while significantly reducing the validation time. The single blind PES provided by EPA (or any other reputable firm) is analyzed with samples of each matrix in a Sample Delivery Group (SDG). A software program (e.g.,PEAC TOOLS, SPS Web or equivalent)is used to determine whether or not the PES results fall within the previously statistically determined acceptance limits ("Action Low" and "Action High")for the Contaminants of Concern (**COC**). The PES results falling within the Action Limits are considered as acceptable results and may be designated as "Passed" analytes, and results of the analytes falling outside the Action Limits are considered as unacceptable and may be designated as "Failed" analytes. In either case ("Passed" Analytes or "Failed" analytes), the associated data is validated according to the Region 2 data validation SOP HW-2 in conjunction with the latest version of the WinCadre QC reports. The following strategy (procedure) is used:

7.2 "Passed" COC

If the COC in an SDG are within statistically generated Action Limits, the data validation is conducted according to QC analyses indicated by check marks (√)in the "Review COC For" column of the Table I. The SDG samples are validated using the Region 2 data validation SOP in conjunction with the latest version of the WinCADRE QC reports. The validation

flags (J, R, U) are applied on Form I's as well on the CADRE Lotus 1,2,3 or XLS spreadsheet. Corrections, if needed, are then made on the Lotus or XLS spreadsheet to ensure that all results on Form I's carry the same data validation and concentration flags as are on the Lotus or XLS Spreadsheet.

7.3 "Failed" COC

If the COC in an SDG are not within the statistically generated Action Limits, the data validation is conducted according to the data validation SOP QC Criteria indicated by check marks (√) in the "Review COC For" column of Table II. The SDG samples are validated using the Region 2 data validation SOP in conjunction with the latest version of the WinCADRE QC reports. The data validation flags (J,R,U) are applied on Form I's as well on the CADRE Lotus 1,2,3 or XLS Spreadsheet. Corrections, if needed, are then made on the Lotus or XLS spreadsheet to ensure that all results on Form I's carry the same data validation and concentration flags as are on the Lotus or XLS Spreadsheet.

7.4 COC "Not Evaluated"

Acceptance limits for the analytes not present/spiked in the PE sample are not provided on the PES Scoring Evaluation Report. Such analytes will be marked as "Not Evaluated" in the PES Evaluation Column. These analytes will be validated much the same way as the "Failed Analytes".

The failed analytes and the analytes not present/spiked in the PE sample require data validation according to the QC criteria specified in Table II, and are identified by the TOPO in the TDF for the Case/SDG.

Table I

Passed PES - All Contaminants of Concern are within the limits
(Action Low ≤ PES Result ≤ Action High)

QC Criteria	Review COC for
Holding Time & Preservation	√
Initial Calibration	
Initial Calibration Verification	
CRQL Standard	√
Blanks-Initial & Continuing	
Preparation Blank	
ICP Interference Check Sample	
Pre- Digestion/Distillation Matrix Spike	
Post Digestion Spike	
Laboratory Duplicate	
Field Duplicates Comparison	√
Lab Control Sample	
ICP Serial Dilution	
Field Blank Contamination	√
Percent Solids	√
Transcription/Computation Check	
Raw Data	
Total vs. Dissolved Concentrations Comparison	√

- The CSF (Complete SDG File) audit will be completed before the PES validation strategy is applied.
- Comparison of the Lotus or XLS Spreadsheet must be after the PES validation strategy is applied. The Contract
- Compliance can be checked after the PES validation strategy is applied.

Table II

Failed PES - Contaminants of Concern are not within the limits
(PES Result ≤ Action Low, PES Result ≥ Action High **OR** The Limits Not Established)

QC Criteria	Review COC for
Holding Time & Preservation	√
Initial Calibration	
Initial Calibration Verification	
CRQL Standard	√
Blanks-Initial & Continuing	
Preparation Blank	√
ICP Interference Check Sample	
Pre- Digestion/Distillation Matrix Spike	√
Post Digestion Spike	
Laboratory Duplicate	√
Field Duplicates Comparison	√
Lab Control Sample	√
ICP Serial Dilution	√
Field Blank Contamination	√
Percent Solids	√
Transcription/Computation Check	√
Raw Data	
Total vs. Dissolved Concentrations Comparison	√

- The CSF (Complete SDG File) audit will be completed before the PES validation strategy is applied.
- Comparison of the Lotus or XLS Spreadsheet must be after the PES validation strategy is applied.
- The Contract Compliance can be checked after the PES validation strategy is applied.

8.0 **Sampling Trip Report**

The sampler prepares a Sampling Trip Report for each sampling event and sends it to the RSCC. The report provides details of all activities performed for each sampling event on the Superfund site. It also lists the field QC samples such as Field Duplicates, Field/Rinse Blanks, sampling time and date for each sample, and samples associated with each field/rinse blank. The validator must use this information to evaluate the Field Duplicate pairs as well as the samples associated with contaminated Field/Rinse Blanks.

9.0 **Telephone Record Log (Appendix A.3)**

A Telephone Record Log (Appendix A.3) must be written by the data validator when a deliverable is missing or a clarification is needed about a lab procedure. The data validator should outline a basic profile of the Case on the Telephone Record Log Form, clearly indicating the reason(s) for inquiry and forward this Form to CLP PO/TOPO who will contact the lab to receive the missing document or information. The original Telephone Record Log is kept in the data package and a copy attached to the Data Review Narrative.

10.0 **Request for Re-Analysis (Appendix A.6)**

Data validator must note all items of contract non-compliance in the Data Review Narrative. If holding times and sample storage times have not been exceeded, the Project Officer (PO) may request re-analysis if items of non-compliance are critical to data assessment. Requests are to be made on "CLP Re-Analysis Request/Approval Record" form (Appendix A.4).

11.0 **CLP Data Assessment Summary Form (Appendix A.7)**

Fill in the total number of analytes performed by different methods and the number of analytes rejected (R) or flagged (J) as estimated due to corresponding quality control criteria. Place an "X" in boxes wherever analyses were not performed, or criteria do not apply.

12.0 **Data Review Log:**

It is recommended that the data validator maintain a log of the reviews completed to document:

a. Case number
b. SDG # (s)
c. number of samples
d. matrix of samples
e. contract laboratory
f. site name
g. start-date of the data case review
h. completion-date of the data case review
i. actual hours spent
j. reviewer's signature

13.0 **Record of Communication** -
This is a Regional document prepared and provided by the RSCC for each data package. The ROC indicates the Case #, site name, samples and sample matrix and the laboratory name. The presence of a ROC in a data package is an indication that the package has been reviewed by the RSCC for completeness and is ready for data validation.

14.0 **Forwarded Paperwork**
Upon completion of review, the following are to be forwarded to EPA for final review:

a. Data package
b. Completed data assessment checklist (Appendix A.1,original)
c. Original and a copy of completed data review narrative Appendix A.2)
d. CLASS Contract Compliance Screening (CCS) report
e. Telephone Record Log (Appendix A.3)
f. Field Duplicates Form (Appendix A.4)
g. Total/Dissolved Concentrations Form (Appendix A.5)
h. CLP Re-analysis Request/Approval Record Form (Appendix A.6)
i. Data Assessment Summary Form (Appendix A.7)
j. CADRE Spreadsheet on a computer diskette.

ACRONYMS

AA	Atomic Absorption
AOC	Analytical Operations/Data Quality Center
CADRE	Computer-Aided Data Review and Evaluation
CCB	Continuing Calibration Blank
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CLP	Contract Laboratory Program
CO	Contracting Officer
COC	Contaminants of Concern
CRI	CRQL Check Standard
CRQL	Contract Required Quantitation Limit
CSF	Complete SDG File
CVAA	Cold Vapor AA
DART	Data Assessment Rapid Transmittal
DAT	Data Assessment Tool
DF	Dilution Factor
DQO	Data Quality Objective
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
ICS	Interference Check Sample
ICV	Initial Calibration Verification
LCS	Laboratory Control Sample
LRS	Linear Range Sample
MDL	Method Detection Limit
NIST	National Institute of Standards and Technology
OERR	Office of Emergency and Remedial Response
OSWER	Office of Solid Waste and Emergency Response
PB	Preparation Blank
PE	Performance Evaluation
%D	Percent Difference
%R	Percent Recovery
%RI	Percent Relative Intensity
%RSD	Percent Relative Standard Deviation
%S	Percent Solids
PO	Project Officer
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RPD	Relative Percent Difference
RSCC	Regional Sample Control Center
SDG	Sample Delivery Group
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
TAL	Target Analyze List

Inorganic Target Analyze List And Contract Required Quantitation Limits (CRQLs)

Analyze	CAS Number	ICP-AES CRQL Water Ug/L	ICP-AES CRQL Soil mg/kg	ICP-MS CRQL Water Ug/L
Aluminum	7429-90-5	200	20	---
Antimony	7440-36-0	60	6	2
Arsenic	7440-38-2	10	1	1
Barium	7440-39-3	200	20	10
Beryllium	7440-41-7	5	0.5	1
Cadmium	7440-43-9	5	0.5	1
Calcium	7440-70-2	5000	500	-----
Chromium	7440-47-3	10	1	2
Cobalt	7440-48-4	50	5	1
Copper	7440-50-8	25	2.5	2
Iron	7439-89-6	100	10	----
Lead	7439-92-1	10	1	1
Magnesium	7439-95-4	5000	500	-----
Manganese	7439-96-5	15	1.5	1
Mercury	7439-97-6	0.2	0.1	---
Nickel	7440-02-0	40	4	1
Potassium	7440-09-7	5000	500	-----
Selenium	7782-49-2	35	3.5	5
Silver	7440-22-4	10	1	1
Sodium	7440-23-5	5000	500	-----
Thallium	7440-28-0	25	2.5	1
Vanadium	7440-62-2	50	5	1
Zinc	7440-66-6	60	6	2
Cyanide	57-12-5	10	2.5	--

--

Site:

Case #:

SDG #:

Samples: Soil Water

		YES	NO	N/A
A.1.1	**Contract Compliance Screening Report** Present?	[___]	___	___
	ACTION: If no, contact RSCC/PO.			
A.1.2	**Record of Communication (from RSCC)**			
	Present?	[___]	___	___
	ACTION: If no, request from the RSCC.			
A.1.3	**Sampling Trip Report**			
	Present and complete?	[___]	___	___
	ACTION: If no, contact RSCC/PO.			
A.1.4	**Chain of Custody/Sample Traffic Report**			
	Present?	[___]	___	___
	Legible?	[___]	___	___
	Signature of sample custodian present?	[___]	___	___
	ACTION: If no, contact RSCC/WAM/PO.			
A.1.5	**Cover Page**			
	Present?	[___]	___	___
	Is the Cover Page properly filled in and the verbatim signed by the lab manager or the manager's designee?	[___]	___	___
	Do the sample identification numbers on the Cover Page agree with sample Identification numbers on:			
	(a) Traffic Report Sheet?	[___]	___	___
	(b) Form I's?	[___]	___	___
	Is the number of samples on the Cover Page the same as the number of			

		YES	NO	N/A
	samples on the Traffic Report sheet and the Regional Record of Communication (ROC) for the data Case?	[___]	___	___

ACTION:
If no for any of the above, prepare Telephone Record Log and contact RSCC/PO for re-submittal of the corrected Cover Page from the laboratory.

A.1.6 **SDG Narrative, DC-1 & DC-2 Form**

Is the SDG Narrative present?	[___]	___	___
Is Sample Log-In Sheet(Form DC-1) present and complete?	[___]	___	___
Is Complete SDG Inventory Sheet(Form DC-2) present and complete?	[___]	___	___

ACTION:
If no, write in the Contract-Problems/ Non-Compliance Section of the Data Review Narrative.

A.1.7 **Form I to XV**

A.1.7.1 Are all the Form I through Form XV labeled with:

Laboratory Name?	[___]	___	___
Laboratory Code?	[___]	___	___
RAS/Non-RAS Case No.?	[___]	___	___
SDG No.?	[___]	___	___
Contract No.?	[___]	___	___

ACTION:
If no for any of the above, note under Contract Problem/Non-Compliance Section of the "Data Review Narrative" and contact PO for corrected Form(s) from the laboratory.

		YES	NO	N/A
A.1.7.2	After comparing values on Forms I-IX against the raw data, do any computation/ transcription errors exceed 10% of the reported values on the Forms for:			
	(a) all analytes analyzed by ICP-AES?	___	[___]	___
	(b) all analytes analyzed by ICP-MS?	___	[___]	___
	(c) Mercury?	___	[___]	___
	(d) Cyanide?	___	[___]	___

ACTION:
If yes, prepare Telephone Record Log and contact CLP PO/TOPO for the corrected data from the laboratory.

A.1.8 **Raw Data**

Data shall not be validated without the hard/electronic copies of the associated raw data for samples and QC samples.

A.1.8.1 **Digestion/Distillation Log**

Digestion Log for ICP-AES (Form XII)present?	[___]	___	___
Digestion Log for ICP-MS (Form XII) present?	[___]	___	___
Digestion Log for mercury (Form XII) present?	[___]	___	___
Distillation Log for cyanide (Form XII) present?	[___]	___	___
Are pH values for metals and cyanide reported for each aqueous sample?	[___]	___	___
Are percent solids calculations present for soils/sediments?	[___]	___	___
Are preparation dates present on the sample preparation logs/bench sheets?	[___]	___	___

		YES	NO	N/A
	NOTE: Digestion/Distillation log must include weights, volumes, and dilutions used to obtain the reported results.			
A.1.8.2	Is the analytical instrument real-time printouts present for:			
	ICP-AES?	[___]	___	___
	ICP-MS?	[___]	___	___
	Mercury?	[___]	___	___
	Cyanide?	[___]	___	___
	Are all laboratory bench sheets and instrument raw data printouts necessary to support all sample analyses and QC operations:			
	Legible?	[___]	___	___
	Properly labeled?	[___]	___	___
	Are all field samples, QC samples and field QC samples present on:			
	Digestion/Distillation log?	[___]	___	___
	Instrument Printouts?	[___]	___	___

ACTION:
If no for any of the above questions in Section A.1.8.1 and Section A.1.8.2, write Telephone Record Log and contact TOPO/PO for re-submittal from the laboratory.

A.1.9 **Technical Holding Times**: (Aqueous and soil samples)
(Examine sample Traffic Reports and digestion/distillation logs to determine the holding time from the sample collection date to the sample preparation date.)

A.1.9.1	Cyanide distillation(14 days)exceeded?	___	[___]	___
	Mercury analysis(28 days) exceeded?	___	[___]	___
	Other Metals analysis(180 days)exceeded?	___	[___]	___

		YES	NO	N/A

ACTION:
If yes, reject (R) and red-line non-detects and flag as estimated (J) results ≥ MDL even if sample(s) was preserved properly.

NOTE:
In addition to qualifying the data, a list of all samples and analytes which exceeded the holding times must be prepared. Report for each sample the number of days that were exceeded. (Subtract the sample collection date from the sample preparation date). Attach this list to the data review narrative.

A.1.9.2 Is pH of aqueous samples for:

Metals Analysis	≤ 2?	[___]	___	___
Cyanide Analysis	≥ 12?	[___]	___	___

ACTION:
If no for any of the above, flag non-detects as "R" and detects as "J".

A.1.9.3 Is the cooler temperature ≤ 10 C°? [___] ___ ___

ACTION:
If cooler temperature is >10 °C , flag non-detects as "UJ" and detects as "J".

A.1.10 **Final Data Correctness - Form I**

A.1.10.1 Are Form I's for all samples present and complete? [___] ___ ___

ACTION:
If no, prepare Telephone Record Log and contact CLP PO/TOPO for submittal from the laboratory.

A.1.10.2 Verify there are no calculation and transcription errors in the results reported on Form I's. Circle on each Form I all results that are incorrect.

		YES	NO	N/A
	Is the calculation error less than 10% of the correct result?	[___]	___	___
	Are results on Form I's reported in correct units (ug/L for aqueous and MG/KG for soils)?	[___]	___	___
	Are results on Form I'S reported by correct significant figures?	[___]	___	___
	Are soil sample results on Form I's corrected for percent solids?	[___]	___	___
	Are all "less than MDL" values reported by the CRQLs and coded with "U"?	[___]	___	___
	Are values less than the CRQLs but greater than or equal to the MDLs flagged with "J"?	[___]	___	___
	Are appropriate contractual quality control and Method qualifiers used?	[___]	___	___

ACTION:
If no for any of the above questions, prepare Telephone Record Log, and contact CLP PO/TOPO for corrected data.

A.1.10.3	Do EPA sample identification numbers and the corresponding laboratory sample identification numbers match on the Cover Page, Form I's and in the raw data?	[___]	___	___
	Was a brief physical description of the samples before and after digestion given on the Form I's?	[___]	___	___
	Was any sample result outside the mercury/cyanide calibration range or the ICP-AES/ICP-MS linear range diluted and noted on the Form I?	[___]	___	___

ACTION:
If no for any of the above, note under the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

		YES	NO	N/A
A.1.11	**Initial Calibration**			
A.1.11.1	Is a record of at least 2 point (A blank and a standard) calibration present for ICP-AES analysis?	[___]	___	___
	Is a record of at least 2 point (a blank and a standard) calibration present for ICP-MS analysis?	[___]	___	___
	Is a record of at least 5 point calibration (a blank & 4 standards) present for Hg analysis?	[___]	___	___
	Is a record of at least 4 point calibration (a blank & 4 standards) present for cyanide?	[___]	___	___
	ACTION: If incomplete or no initial calibration was performed, reject (R) and red-line the associated data (detects & non-detects).			
	Is one initial calibration standard at the CRQL level for cyanide and mercury?	[___]	___	___
	ACTION: If no, write in the Contract Problem/ Non-Compliance Section of the Data Review Narrative.			
A.1.11.2	Is the curve correlation coefficient $\geq$ 0.995 for:			
	Mercury Analysis?	[___]	___	___
	Cyanide Analysis?	[___]	___	___
	ICP-AES (more than 2 point Calib.)?	[___]	___	___
	ICP-MS (more than 2 point calib.)?	[___]	___	___
	ACTION: If no, qualify the associated sample results $\geq$ MDL as estimated "J" and non-detects as "UJ".			

NOTE:
The correlation coefficient shall be calculated by the data validator using standard concentrations and the

		YES	NO	N/A
	corresponding instrument response (e.g. absorbance, peak area, peak height, etc.).			
A.1.12	**Initial and Continuing Calibration Verification- Form IIA**			
A.1.12.1	Present and complete for every metal and cyanide?	[___]	___	___
	Present and complete for ICP-AES and ICP-MS when both these methods were used for the same analyte?	[___]	___	___
	ACTION: If no for any of the above, prepare a Telephone Record Log and contact PO/TOPO for re-submittal from the laboratory.			
A.1.12.2	Was a Continuing Calibration Verification performed every 10 samples or every 2 hours whichever is more frequent?	[___]	___	___
	ACTION: If no for any of the above, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative.			
A.1.12.3	Was an ICV or a mid-range standard distilled and analyzed with each batch of cyanide samples?	[___]	___	___
	ACTION: If no for any of the above, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative and qualify results $\geq$ MDL as estimated (J).			
A.1.12.2	Circle on each Form IIA all percent recoveries that are outside the contract windows.			
	Are ICV/CCVs within control limits for:			
	Metals - 90-110%R?	[___]	___	___
	Hg - 80-120%R?	[___]	___	___
	Cyanide - 85-115%R?	[___]	___	___

		YES	NO	N/A

ACTION:
If no, qualify all samples between a previous technically acceptable CCV standard and a subsequent technically acceptable CCV standard as follows as follows:

Qualify as estimated (J) all detects and non-detects, if the ICV/CCV %R is between 75-89%(65-79% for Hg; 70-84% for CN). Qualify only positive results(≥ MDL) as "J" if the ICV/CCV %R is between 111-125%(121-135% for Hg;116-130% for CN). Reject (R) and red-line only detects if the recovery is greater than 125% (135% for Hg; 130% for CN). Reject (R) and red-line all associated results (hits and non-detects)if the recovery is less than 75%(65% for Hg;70% for CN).

NOTE:
For ICV that does not fall within the acceptance limits, qualify all samples reported from the analytical run.

A.1.12.3 Was the distilled ICV or mid-range standard for cyanide within acceptance limits (85-115%)? [___] ___ ___

ACTION:
If no, Qualify all cyanide results ≥ MDL as "J".

A.1.13 **CRQL Standard Analysis - Form IIB**

A.1.13.1 For each ICP-AES run, was a CRI (CRQL or MDL when MDL > CRQL) standard analyzed? [___] ___ ___
(Note:CRI is not required for Al, Ba, Ca, Fe, Mg, Na and K.)

For each ICP-MS run, was a CRI (CRQL or MDL when MDL > CRQL) standard analyzed for each mass/isotope used for the analysis? [___] ___ ___

For each mercury run, was a CRQL standard analyzed? [___] ___ ___

For each cyanide run, was a CRQL standard analyzed? [___] ___ ___

		YES	NO	N/A

ACTION:
If no for any of the above, write this deficiency in the Contract Problems/ Non-Compliance Section of the Data Review Narrative, inform CLP PO and flag results in the affected ranges (detects <2xCRQL)as J and non-detects UJ.

The affected ranges are:
ICP-AES Analysis - *True Value ± CRQL
ICP-MS Analysis - *True Value ± CRQL
Mercury Analysis - *True Value ± CRQL
Cyanide Analysis - *True Value ± CRQL
*** True value of the CRQL Standard**

A.1.13.2 Was a CRQL standard analyzed after the ICV/ICB, before the final CCV/CCB and once every 20 analytical samples in the analytical run for each analysis? [___] ___ ___

ACTION:
If no, write in the Contract Problem/ Non-Compliance Section of the "Data Review Narrative".

A.1.13.3 Circle on each Form IIB all percent recoveries that are outside the acceptance windows.

Is the CRQL standard within control limits for:

Metals(ICP-AES/ICP-MS)- 70 - 130%? [___] ___ ___

Mercury- 70 - 130%? [___] ___ ___

Cyanide - 70 - 130%? [___] ___ ___

ACTION:
If no, flag detects <2xCRQL as "J" and non-detects as "UJ" if the CRQL standard recovery is between 50-69%. Flag(J) only detects <2xCRQL if the recovery is between 131% and ≤180%. If the recovery is less than 50%, reject(R) and red-line non-detects and detects < 2xCRQL, and flag (J) detects between 2xCRQL and ICV/CCV. Reject and red-line only detects <2xCRQL and flag (J)detects ≥ 2xCRQL but < ICV/CCV if the recovery is > 180%.

		YES	NO	N/A

NOTE:
1. Qualify all field samples analyzed between a previous technically acceptable analysis of the CRQL standard and a subsequent acceptable analysis of the CRQL standard
2. Flag (J) or reject (R) only the final sample results on Form I's when **Sample raw data** are within the affected ranges and the CRQL standard is outside the acceptance windows.
3. The samples and the CRQL standard must be analyzed in the same analytical run.

A.1.14 **Initial and Continuing Calibration Blanks - Form III**

A.1.14.1	Present and complete for all the instruments used for the metals and cyanide analyses?	[___]	___	___
	Was an initial Calibration Blank analyzed after ICV?	[___]	___	___
	Was a continuing Calibration Blank analyzed after every CCV and every 10 samples or every 2 hours, whichever is more frequent?	[___]	___	___
	Were the ICB & CCB values ≥ MDL but < CRQL reported on Form III and flagged "J" by using MDLs from direct analysis(Preparation Method "NP1")? (Check Form III against the raw data)	[___]	___	___

ACTION:
If no, inform CLP PO/TOPO and make a note in the Contract-Problems/Non-Compliance Section of the "Data Review Narrative".

A.1.14.2 Circle with red pencil on each Form III all Calib. Blank values that are:

≥ MDL but ≤ CRQL

> CRQL

A.1.14.2.1	When MDL < CRQL, is any Calib. Blank value ≥ MDL but ≤ CRQL?	___	[___]	___

ACTION:
If yes, change sample results ≥ MDL but ≤ CRQL to the CRQL with a "U". Do not qualify non-detects.

		YES	NO	N/A
A.1.14.2.2	When MDL < CRQL, is any Calib. Blank value > CRQL?	___	[___]	___

ACTION:
If yes, reject (R) and red line the associated sample results > CRQL but <ICB/CCB Blank Result. Flag as "J" detects > ICB/CCB blank value but < 10xICB/CCB value. Change the sample results ≥ MDL but ≤ the CRQL to CRQL with a "U".

A.1.14.2.3	Is any Calibration Blank value below the negative CRQL?	___	[___]	___

ACTION:
If yes, flag (J) as estimated all associated sample results ≥ CRQL but <10xCRQL.

NOTE:
1. For ICB that does not meet the technical QC Criteria, apply the action to all samples reported from the analytical run.
2. For CCBs that do not meet the technical QC criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of CCB and a subsequent technically acceptable analysis of the CCB in the analytical run.,

A.1.15 Preparation Blank - FORM III

NOTE:The Preparation Blank for mercury is the same as the calibration blank.

A.1.15.1	Was one Preparation Blank prepared with and analyzed for:			
	Each Sample Delivery Group (SDG)?	[___]	___	___
	Each batch of the SDG samples digested/distilled?	[___]	___	___
	Each matrix type?	[___]	___	___
	All instruments used for metals and cyanide analyses?	[___]	___	___

	YES	NO	N/A

ACTION:
If no for any of the above, flag as estimated (J) all the associated positive data <10xMDL for which the Preparation Blank was not analyzed.

NOTE:
If only one blank was analyzed for more than 20 samples, then the first 20 samples analyzed are not estimated(J),but all additional samples must be qualified (J).

A.1.15.2 Circle with red pencil on each Form III all Prep. Blank values that are:

≥ MDL but ≤ CRQL, and

> CRQL

A.1.15.2.1 When MDL < CRQL, is any preparation blank value ≥ MDL but ≤ CRQL? ___ [___] ___

ACTION:
If yes, change sample result ≥ MDL but ≤ CRQL to CRQL with a "U".

A.1.15.2.2 When the MDL ≤ CRQL, is any Preparation Blank value greater than its CRQL? ___ [___] ___

If yes, is the Prep. Blank value greater than the value of the associated Field Blank collected and analyzed with the SDG samples? ___ [___] ___

If yes, is the lowest concentration of that analyte in the associated samples less than 10 times the Preparation Blank value? ___ [___] ___

ACTION:
If yes, reject (R) and red-line all associated sample results greater than the CRQL but less than the Prep.Blank value. Flag as "J" detects > Prep. Blank value but <10xPrep.Blank. If the sample result ≥ MDL but ≤ CRQL, replace it with CRQL-U.

If the Prep. Blank value is less than the same

		YES	NO	N/A
	analyte value in the Field Blank, do not qualify the sample results due to the Prep. Blank criteria.			
	NOTE: Convert soil sample result to mg/Kg on wet weight basis to compare with the soil Prep. Blank result on Form III.			
A.1.15.2.3	Is the Prep. Blank concentration below the negative CRQL?	___	[___]	___
	ACTION: If yes, flag (J) all associated sample results less than 10xCRQL. Qualify non-detects as estimated (UJ).			
A.1.15.2.4	When the MDL is greater than the CRQL, is the preparation blank concentration on Form III greater than two times the MDL?	___	[___]	___
	ACTION: If yes, reject (R) and red-line all positive sample results with sample raw data less than 10 times the Preparation Blank value.			
A.1.16	**ICP-AES/ICP-MS Interference Check Sample (ICS)- Form IV**			
	NOTE: Not required for CN, Hg, Al, Ca, Fe and Mg.			
A.1.16.1	Present and complete?	[___]	___	___
	Was ICS analyzed at the beginning and end of each analytical run, and once for every 20 analytical samples?	[___]	___	___
	Was ICS analyzed at the beginning of the ICP-MS analytical run?	[___]	___	___
	ACTION: If no, flag as estimated (J) all sample results.			

	YES	NO	N/A

A.1.16.2 **ICP-AES Method**

A.1.16.2.1 **ICSA Solution:**

For ICP-AES, are the ICSA "Found" analyte values within the control limits ± of CRQL of the true/established mean value? [___] ___ ___

If no for any of the above, is the sample concentration of Al, Ca, Fe, or Mg in the same units (ug/L or MG/KG) greater than or equal to its respective concentration in the ICSA Solution on Form IV? [___] ___ ___

ACTION:

If yes, apply the following action to all samples analyzed between a previous technically acceptable analysis of the ICS and a subsequent technically acceptable analysis of the ICS in the analytical run:

Flag (J) as estimated only sample results ≥MDL for which the ICSA "Found" value is greater than (True value+CRQL). Do not qualify non-detects. If the ICSA "Found" value is less than (True value-CRQL), flag non-detects as "UJ" and detects as "J".

A.1.16.2.3 **ICSAB Solution**

For ICP-AES, are all analyte results in ICSAB within the control limits of 80-120 of the true/established mean value? [___] ___ ___

If no for any of the above, is the sample concentration of Al, Ca, Fe, or Mg in the same units (ug/L or MG/KG) greater than or equal to its respective concentration in the ICSAB Solution on Form IV? [___] ___ ___

ACTION:

If yes, apply the following action to all samples analyzed between a previous technically acceptable analysis of the ICS and a subsequent technically acceptable analysis of the ICS in the analytical run:

Flag (J) as estimated those associated sample results ≥ MDL for which the ICSAB analyte recovery is greater than 120% but ≤ 150%. If the ICSAB recovery falls within

		YES	NO	N/A
	50-79%, qualify sample results ≥ MDL as "J" and non-detects as "UJ". Reject (R) and red-line all sample results (detects & non-detects) for which the ICSAB analyte recovery is less than 50%. If the recovery is above 150%, reject (R) and red-line only positive results.			
A.1.16.3	**ICP-MS Method**			
A.1.16.3.1	**ICSA Solution:** For ICP-MS, are the ICSA "Found" analyte values within the control limits of ±CRQL of the true/established mean value?	[___]	___	___
	ACTION: If no, apply the following action to all samples reported from the analytical run: Flag (J) as estimated only sample results ≥ MDL if the ICSA "Found" value is greater than (True value+CRQL). Do not qualify non-detects. If the ICSA "Found" value is less than (True value-CRQL), flag the associated sample detects as "J" and non-detects as "UJ".			
A.1.16.3.3	**ICSAB Solution** For ICP-MS, are all analyte results in ICSAB within the control limits of 80-120% of the true/established mean value, whichever is greater?	[___]	___	___
	ACTION: If no, apply the following action to all samples reported from the analytical run: Flag (J) as estimated those associated sample results ≥ MDL for which the ICSAB analyte recovery is greater than 120% but ≤ 150%. If the ICSAB recovery falls within 50-79% flag (J) as estimated the associated sample results ≥ MDL. Reject (R) and red-line those all sample detects and non-detects for which the ICSAB analyte recovery is less than 50%. If the recovery is above 150%,reject (R) and red-line only detects (≥ MDL).			
A.1.17	**Spiked Sample Recovery: Pre-Digestion/Pre-Distillation)-Form V A** **Note**:Not required for Ca,Mg,K,and Na(both matrices);Al and Fe (soil only)			
A.1.17.1	Was Matrix Spike analysis performed: For each matrix type?	[___]	___	___

		YES	NO	N/A
	For each SDG?	[___]	___	___
	On one of the SDG samples?	[___]	___	___
	For each concentration range (i.e.,low, med., high)?	[___]	___	___
	For each analytical Method (ICP-AES,ICP-MS, Hg, CN)used?	[___]	___	___
	Was a spiked sample prepared and analyzed with the SDG samples?	[___]	___	___
	ACTION: If no for any of the above, flag as estimated(J)all the positive data for which a spiked sample was not analyzed.			
	NOTE: If more than one spiked sample were analyzed for one SDG, then qualify the associated data based on the worst spiked sample analysis.			
A.1.17.2	Was a field blank or PE sample used for the spiked sample analysis?	___	[___]	___
	ACTION: If yes, flag (J) as estimated positive data of the associated SDG samples for which field blank or PE sample was used for the spiked sample analysis.			
A.1.17.3	Circle on each Form VA all spike recoveries that are outside the control limits (75-125%) that have sample concentrations less than four times the added spike concentrations.			
	Are all recoveries within the control limits when sample concentrations are less than or equal to four times the spike concentrations?	[___]	___	___ ___
	NOTE: Disregard the out of control spike recoveries for analytes whose concentrations are greater than or equal to four times the spike added.			
	Are results outside the control limits			

	YES	NO	N/A
(75-125%) flagged with Lab Qualifier "N" on Form I's and Form VA?	[___]	___	___

ACTION:
If no for any of the above, write in the Contract - Problems/Non-Compliance Section of the Data Review Narrative.

A.1.17.4 **Aqueous**

Are any spike recoveries:

(a) less than 30%?	___	[___]	___
(b) between 30-74%?	___	[___]	___
(c) between 126-150%?	___	[___]	___
(d) greater than 150%?	___	[___]	___

ACTION:
If the matrix spike recovery is less than 30%, reject (R) and red-line all associated aqueous data (detects & non-detects). If between 30-74%, qualify all associated aqueous data ≥ MDL as "J" and non-detects as "UJ". If between 126-150%, flag (J) all data ≥ MDL as "J". If greater than 150%, reject (R) and red-line all associated data ≥ MDL.

(**NOTE:** Replace "N" with "J", "R" as appropriate.)

A.1.17.5 **Soil/Sediment**

Are any spike recoveries:

(a) less than 10%?	___	[___]	___
(b) between 10-74%?	___	[___]	___
(c) between 126-200%?	___	[___]	___
(d) greater than 200%?	___	[___]	___

ACTION:
If yes for any of the above, proceed as follows:

If the matrix spike recovery is less than 10%, reject (R) and red-line all associated data (detects & non-detects); if between 10-74%, qualify all associated data ≥ MDL as "J" and non-detects as "UJ";

		YES	NO	N/A
	if between 126-200%, flag (J) all associated data ≥ MDL as "J" If greater than 200%, reject (R) and red-line all associated data ≥ MDL. (**NOTE:**Replace "N" with "J" or "R" as appropriate.)			
A.1.18	**Lab Duplicates) - Form VI**			
A.1.18.1	Was the lab duplicate analysis performed:			
	For each SDG?	[___]	___	___
	On one of the SDG samples?	[___]	___	___
	For each matrix type?	[___]	___	___
	For each concentration range (low or med.)?	[___]	___	___
	For each analytical Method (ICP-AES/ICP-MS,Hg,CN)Used?	[___]	___	___
	Was a lab duplicate prepared and analyzed with the SDG samples?	[___]	___	___
	ACTION: If no for any of the above, flag (J) as estimated all the SDG sample results (detects & non-detects) for which the lab duplicate analysis was not performed.			
	NOTE: If more than one lab duplicate sample were analyzed for an SDG, then qualify the associated samples based on the worst lab duplicate analysis.			
A.1.18.2	Was a Field Blank or PE sample used for the Lab Duplicate analysis?	___	[___]	___
	ACTION: If yes, flag as estimated (J) all SDG sample results (hits & non-detects) for which Field Blank or PE sample was used for duplicate analysis.			
A.1.18.3	Circle on each Form VI all values that are:			
	RPD > 20%, or			
	Absolute Difference > CRQL			
	Are all values within control limits (RPD ≤ 20% or absolute			

	YES	NO	N/A
difference ≤ ±CRQL)?	[___]	___	___
If no, are all results outside the control limits flagged with an "*" (Lab Qualifier)on Form VI and on all Form I's?	[___]	___	___

ACTION:
If no, write in the Contract-Problems/ Non-Compliance Section of the Data Review Narrative.

NOTE:
The laboratory is not required to report on Form VI the RPD when both values are non-detects.

A.1.18.4 **Aqueous**

A.1.18.4.1 When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),

is any RPD > 20% but < 100%?	___	[___]	___
is any RPD ≥ 100%?	___	[___]	___

ACTION:
If the RPD is > 20% but < 100%, flag (J) as estimated the associated sample data ≥ CRQL. If the RPD is ≥ 100%, reject (R) and red-line the associated sample data ≥ CRQL.

(**NOTE:**Replace "*" with "J" or "R" as appropriate.)

A.1.18.4.2 When the sample and/or duplicate value <5xCRQL (substitute MDL for CRQL when MDL >CRQL), is the absolute difference between sample and duplicate values:

> ± CRQL?	___	[___]	___
> ± 2xCRQL?	___	[___]	___

ACTION:
If the absolute difference is > CRQL, flag as estimated all the associated sample results ≥ MDL but < 5xCRQL as "J" and non-detects as "UJ". If the absolute difference is > 2xCRQL, reject (R) and

	YES	NO	N/A

red-line all the associated non-detects and detects ≥ MDL but < 5xCRQL.

NOTE:
1. Replace "*" with "J", "UJ" or "R" as appropriate.)
2. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this difference to qualify sample results.

A.1.18.5 **Soil/Sediment**

A.1.18.5.1 When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),

is any RPD ≥ 35% but < 120%?	___	[___]	___
is any RPD ≥ 120%?	___	[___]	___

ACTION:
If the RPD is ≥ 35% and < 120%, flag (J) as estimated the associated sample data ≥ CRQL. If the RPD is ≥ 120%, reject (R)and red-line the associated sample data ≥ CRQL.

A.1.18.5.2 When the sample and/or duplicate value <5xCRQL(substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and duplicate:

> ± 2 x CRQL?	___	[___]	___
> ± 4 x CRQL	___	[___]	___

ACTION:
If the absolute difference is > 2 x CRQL, flag all the associated sample results ≥ MDL but < 5xCRQL as "J" and non-detects as "UJ". If the absolute difference is > 4xCRQL, reject (R) and red-line all the associated non-detects and detects ≥ MDL but <5xCRQL.

NOTE:
1. Replace "*" with "J", "UJ" or "R" as appropriate.)
2. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this difference to qualify sample results.

		YES	NO	N/A
A.1.19	**Field Duplicates**			
	Aqueous Field Duplicates			
A.1.19.1	Was an aqueous Field Duplicate pair collected and analyzed? (Check Sampling Trip Report)	[___]	___	___

ACTION:
If yes, prepare a Form (Appendix A.4) for each aqueous Field Duplicate pair. Report the sample and Field Duplicate results on Appendix A.4 from their respective Form I's. Calculate and report RPD on Appendix A.4 when sample and its Field Duplicate values are both > 5xCRQL. Calculate and report the absolute difference on Appendix A.4 when at least one value (sample or duplicate) is <5xCRQL. Evaluate the aqueous Field Duplicate analysis in accordance with the QC criteria stated in Sections A.1.19.2 and A.1.19.3.

NOTE:
1. Do not transfer "*" from Form I's to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when MDL > CRQL.
4. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this the criteria to qualify the results.

A.1.19.2 Circle all values on the Form (Appendix A.4) for Field Duplicates that have:

RPD $\geq$ 20% or

Difference > $\pm$ CRQL

When sample and duplicate values are both $\geq$5xCRQL (substitute MDL for CRQL when MDL > CRQL),

is any RPD $\geq$ 20%?	___	[___]	___
is any RPD $\geq$ 100%?	___	[___]	___

ACTION:
If the RPD is >20% but < 100%, flag (J) only the associated sample and its Field Duplicate results $\geq$ CRQL. If the RPD is $\geq$ 100%, reject(R) and red-line only the associated sample and its Field Duplicate result $\geq$ CRQL.

		YES	NO	N/A
A.1.19.3	When the sample and/or duplicate value(s) <5xCRQL (substitute MDL for CRQL when MDL >CRQL), is the absolute difference between sample and duplicate:			
	> ± CRQL?	___	[___]	___
	> ± 2 x CRQL?	___	[___]	___

ACTION:
If the absolute difference is > CRQL, flag detects ≥ MDL but < 5xCRQL as "J" and non-detects as "UJ". If the difference is > 2xCRQL, reject (R) and red-line non-detects and results ≥ MDL but <5xCRQL of the sample and its Field Duplicate.

Soil/Sediment Field Duplicates

A.1.19.4	Was a soil field duplicate pair collected and analyzed? (Check Sampling Trip Report)	[___]	___	___

ACTION:
If yes, for each soil Field Duplicate pair proceed as follows:

Prepare Appendix A.4 for each Field Duplicate pair. Report on Appendix A.4 all sample and its Field Duplicate results in MG/KG from their respective Form I's. Calculate and report RPD when sample and its duplicate values are both greater than 5xCRQL. Calculate and report the absolute difference when at least one value (sample or duplicate) is < 5xCRQL. Evaluate the Field Duplicate analysis in accordance with the QC Criteria stated in Sections A.1.19.5 and A.1.19.6.

NOTE:
1. Do not transfer "*" from Form I's to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when MDL > CRQL.
4. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and apply the criteria to qualify the results.

		YES	NO	N/A
A.1.19.5	Circle on each Appendix A.4 all values that have: RPD ≥ 35%, or Difference > ± 2xCRQL When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),			
	is any RPD ≥ 35% but < 120%?	___	[___]	___
	is any RPD ≥ 120%?	___	[___]	___

ACTION:
If the RPD is ≥ 35% but < 120%, flag only the associated sample and its Field Duplicate results ≥ CRQL as "J". If the RPD is ≥ 120%, reject (R) and red-line only the sample and its Field Duplicate results ≥ CRQL.

A.1.19.6	When the sample and/or duplicate value(s) <5xCRQL (substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and Field Duplicate:			
	> ± 2 x CRQL?	___	[___]	___
	> ± 4 x CRQL?	___	[___]	___

ACTION:
If the absolute difference is > 2xCRQL, flag Sample and its Field Duplicate resuts ≥ MDL but <5xCRQL as "J" and non-detects as "UJ". If the difference is >4xCRQL, reject(R) and red-line non-detects and detects ≥ MDL but <5xCRQL of the sample and its Field Duplicate.

A.1.20 **Laboratory Control Sample (LCS) - Form VII**

A.1.20.1	Was one LCS prepared and analyzed for:			
	Each SDG?	[___]	___	___
	Each matrix type?	[___]	___	___
	Each batch samples digested/distilled?	[___]	___	___ ___

	YES	NO	N/A
For each Method(ICP-AES,ICP-MS,Hg,CN) used?	[___]	___	___
Was an LCS prepared and analyzed with the samples?	[___]	___	___

ACTION:
If no for any of the above, prepare Telephone Record Log and contact CLP PO or TOPO for submittal of the LCS results. Flag (J) as estimated all the data for which an LCS was not analyzed.

NOTE:
If only one LCS was analyzed for more than 20 samples, then the first 20 samples analyzed are not flagged(J), but all additional samples must be qualified (J).

A.1.20.2 **Aqueous LCS**

Circle on each Form VII the LCS percent recoveries outside control limits 80-120%.

NOTE: 1.Use digested ICV as LCS for aqueous mercury
2.Use distilled ICV as LCS for aqueous cyanide

Is any LCS recovery:

Less than 50%?	___	[___]	___
Between 50% and 79%?	___	[___]	___
Between 121% and 150%?	___	[___]	___
Greater than 150%?	___	[___]	___

ACTION:
If the LCS recovery is less than 50%, reject (R) and red-line all associated sample data (detects & non-detects); for a recovery between 50-79%, flag detects as "J" all non-detects as "UJ". if the LCS recovery is between 121-150%, flag only detects as "J". if the recovery is greater than 150%, reject (R) and red-line all detects.

		YES	NO	N/A
A.1.20.3	**Solid LCS** If an analyte's MDL is equal to or greater than the true value of LCS, disregard the "Action" below for that analyte even though the LCS is out of control limits. Is the LCS "Found" value greater than the Upper Control Limit reported on Form VII? **ACTION**: If yes, flag (J) all the associated detects ≥ MDL as estimated (J). Is the LCS "Found" value lower than the Lower Control Limit reported on Form VII? **ACTION**: If yes, flag detects as "J" and non-dectes as "UJ".	___	[___]	___
A.1.21	**ICP-AES/ICP-MS Serial Dilution - Form VIII** **NOTE**:Serial dilution analysis is required only when the initial concentration is equal to or greater than 50 x MDL.			
A.1.21.1	Was a Serial Dilution analysis performed:			
	For each SDG?	[___]	___	___
	On one of the SDG samples?	[___]	___	___
	For each matrix type?	[___]	___	___
	For each concentration range (low or med.)?	[___]	___	___
	Was a Serial Dilution sample analyzed with the SDG samples? **ACTION**: If no for any of the above, flag as estimated (J) detects ≥ MDL of all the SDG samples for which the ICP Serial Dilution Analysis was not performed.	[___]	___	___

		YES	NO	N/A
A.1.21.2	Was a Field Blank or PE sample used for the Serial Dilution Analysis?	___	[___]	___
	ACTION: If yes, flag as estimated (J) detects ≥ MDL of all the SDG samples			
A.1.21.3	Circle on Form VIII the Percent Differences (%D) between sample results and its dilution results that are outside the control limits ± 10% when initial concentrations ≥ 50 x MDLs.			
	Are results outside the control limits flagged with an "E"(Lab Qualifier) on Form VIII and all Form I's?	[___]	___	___
	ACTION: If no, write in the Contract-Problem/ Non-Compliance Section of the Data Review Narrative.			
A.1.21.4	Are any %D values:			
	> 10%?	___	[___]	___
	≥ 100%?	___	[___]	___
	ACTION: If the Percent Difference (%D) is greater than 10%, flag (J) as estimated all associated samples whose **raw data** ≥ MDL; if the %D is ≥ 100%, reject (R) and red-line all associated samples with **raw data** ≥ MDL.			
	(**NOTE**:Replace "E" with "J" or "R" as appropriate.)			
A.1.22	**Total/Dissolved or Inorganic/Total Analytes**			
A.1.22.1	Were any analyses performed for dissolved as well as total analytes on the same sample(s)?	___	[___]	___
	Were any analyses performed for inorganic as well as total analytes on the same sample(s)?	___	[___]	___
	ACTION: If yes, prepare a Form (Appendix A.5) to compare the differences between dissolved (or inorganic)and total analyte concentrations. Compute each			

		YES	NO	N/A
	difference on Appendix A.5 as a percent of the total analyte only when both of the following conditions are fulfilled: (1) The dissolved(or inorganic)concentration is greater than total concentration, and (2) greater than or equal to 5xMDL.			
A.1.22.2	Is any dissolved (or inorganic) concentration greater than its total concentration by more than 20%?	___	[___]	___
A.1.22.3	Is any dissolved(or inorganic) concentration greater than its total concentration by more than 50%?	___	[___]	___
	ACTION: If the percent difference is greater than 20%, flag (J) both dissolved/inorganic and total concentrations as estimated. If the difference is more than 50%, reject (R) and red-line both the values.			
A.1.23	**Field Blank - Form I** **NOTE: Designate "Field Blank" as such on Form I**			
A.1.23.1	Was a Field/Rinsate Bank collected and analyzed with the SDG samples?	[___]	___	___
	If yes, is any Field/Rinsate Blank absolute value of an analyte on Form I greater than its CRQL(or 2xMDL when MDL>CRQL)?	___	[___]	___
	If yes, circle the Field Blank value on Form I that is greater than the CRQL, (or 2 x MDL when MDL > CRQL).			
	Is any Field Blank value greater than CRQL also greater than the Preparation Blank value?	___	[___]	___
	If yes, is the Field Blank value (> CRQL and > the prep. blank value) already rejected due to other QC criteria?	[___]	___	___
	ACTION: If the Field Blank value was not rejected, reject all associated sample data (except			

		YES	NO	N/A
	the Field Blank results)greater than the CRQL but less than the Field Blank value. Reject on Form I's the soil sample results whose raw values in ug/L in the instrument printout are greater than the CRQL but less than the Field Blank value in ug/L. Flag as "J" detects between the Field Blank value and 10xField Blank value. If the sample result ≥ MDL but ≤ CRQL, replace it with CRQL-U.			
	If the Field Blank value is less than the Prep.Blank value, do not qualify the sample results due to the Field Blank criteria.			

NOTE:
1. Field Blank result previously rejected due to other criteria cannot be used to qualify field samples.
2. Do not use Rinsate Blank associated with soils to qualify water samples and vice versa.

A.1.24 **Verification of Instrumental Parameters - Form IX, XA, XB, XI**

A.1.24.1	Is verification report present for:			
	Method Detection Limits (Form IX-Annually)?	[___]	___	___
	ICP-AES Interelement Correction Factors (Form XA & XB -Quarterly)?	[___]	___	___
	ICP-AES & ICP-MS Linear Ranges (Form XI-Quarterly)?	[___]	___	___

ACTION:
If no, contact CLP PO/TOPO for submittal from the laboratory.

A.1.24.2 **Method Detection Limits - Form IX**

A.1.24.2.1	Are MDLs present on Form IX for:			
	All the analytes?	[___]	___	___
	All the instruments used?	[___]	___	___
	Digested and undigested samples and Calib.Blanks?	[___]	___	___
	ICP-AES and ICP-MS when both instruments are used for the same analyte?	[___]	___	___

		YES	NO	N/A
	ACTION: If no for any of the above, prepare Telephone Record Log and contact CLP PO/TOPO for submittal of the MDLs from the laboratory. Report to CLP PO and write in the Contract Problems/ Non-Compliance Section of the Data Review Narrative if the MDL concentration is not less than ½ CRQL.			
A.1.24.2.2	Is MDL greater than the CRQL for any analyte?	___	[___]	___
	If yes,is the analyte concentration on Form I greater than 5 x MDL for the sample analyzed on the instrument whose MDL exceeds CRQL?	[___]	___	___
	ACTION: If no, flag as estimated (J) all values less than five times MDL for the analyte whose MDL exceeds the CRQL.			
A.1.24.3	**Linear Ranges - Form XI**			
A.1.24.3.1	Was any sample result higher than the high linear range for ICP-AES or ICP-MS?	___	[___]	___
	Was any sample result higher than the highest calibration standard for mercury or cyanide?	___	[___]	___
	If yes for any of the above, was the sample diluted to obtain the result reported on Form I?	[___]	___	___
	ACTION: If no, flag (J) as estimated the affected detects (≥ MDL) reported on Form I.			
A.1.25	**ICP-MS Tune Analysis - Form XIV**			
A.1.25.1	Was the ICP-MS instrument tuned prior to calibration?	[___]	___	___
	ACTION: If no, reject (R) and red-line all sample data for which tuning was not performed.			

		YES	NO	N/A
A.1.25.2	Was the tuning solution analyzed or scanned at least five times consecutively?	[___]	___	___
	Were all the required isotopes spanning the analytical range present in the tuning solution?	[___]	___	___
	Was the mass resolution within 0.1 amu for each isotope in the tuning solution?	[___]	___	___
	Was %RSD less than 5% for each isotope of each analyte in the tuning solution?	[___]	___	___

ACTION:
If no for any of the above, qualify all results ≥ MDL associated with that Tune as estimated "J", and all non-detects associated with that Tune as "UJ".

A.1.26 **ICP-MS Internal Standards - Form XV**

A.1.26.1	Were the Internal Standards added to all the samples and all QC samples and calibration standards (except the Tuning Solution)?	[___]	___	___
	Were all the target analyte masses bracketed by the masses of the five internal standards?	[___]	___	___

ACTION:
If none of the Internal Standards was added to the samples, reject (R) and red-line all the associated sample data (detects & non-detects). If internal standards were used but did not cover all the analyte masses, reject (R) and red-line only the analyte results not bracketed by the internal standard masses.

A.1.26.2	Was the intensity of an Internal Standard in each sample within 60-125% of the intensity of the same Internal Standard in the calibration blank?	[___]	___	___

	YES	NO	N/A
If no, was the original sample diluted two fold, Internal Standard added and the sample re-analyzed?	[___]	___	___
Was the %RI for the two fold diluted sample within the acceptance limits (60-125%)?	[___]	___	___

ACTION:
If no for any of the above, flag detects as "J" and non-detects "UJ" of all the analytes with atomic masses between the

atomic mass of the internal standard lighter than the affected internal standard, and the atomic mass of the internal standard heavier than the affected internal standard.

A.1.27 **Percent Solids of Sediments**

A.1.27.1 Are percent solids in sediment(s):

< 50%?	___	[___]	___

ACTION:
If yes, qualify as estimated (J) all detects and non-detects of a sample that has percent solids less than 50%(i.e.,moisture content greater than 50%).

NOTE:
Flag(J) only the sample results that were not previously flagged due to other QC criteria.

Inorganic Data Review Narrative

Case# _____ **Site:** _____ **Matrix: Soil** ____

SDG# _____ **Lab:** _____ **Water** ____

Sampling Team: _____ **Reviewer:** _____ **Other** _____

A.2.1 **Data Validation Flags:**

The following flags may have been applied in red by the data validator and must be considered by the data user.

J - This flag indicates the result qualified as **estimated**

R and Red-Line - A red-line drawn through a sample result indicates **unusable** value. The red-lined data are known to contain significant errors based on documented information and must not be used by the data user.

U - This data validation qualifier is applied to sample results ≥ MDL when associated blank is contaminated

Fully Usable Data - The results that do not carry "J" or "red-line" are fully **usable**.

A.2.2 **Laboratory Qualifiers:**

The CLP laboratory applies a contractual qualifier on all Form I'S and the QC Form when a QC analysis is outside the control limits. These qualifiers are not applied on the Lotus or XLS spreadsheets. These qualifiers and their meanings are as follows:

N: This qualifier indicates the lack of accuracy in the reported result, and is applied when matrix spiked sample recovery is outside the control limits.

E: This qualifier indicates the the presence of interference, and is applied when the ICP serial dilution is outside the control limits.

*: This qualifier indicate the lack of precision , and is pplied on Fom I'S and Form VI when the Lab Duplicate analysis is outside the control limits.

U: This is a concentration qualifier that laboratory applies to a non-detected result which is essentially less than the Method Detection Limit(MDL). A non-detected result of an analyte is indicated by the Contract Required Quantitation Limit (CRQL) of that analyte suffixed with "U".

J: This is also a concentration qualifier that laboratory applies to a positive result below the CRQL.

NOTE: The laboratory qualifiers are crossed out and replaced with the appropriate data validation qualifiers (J, R or U) by the data validator.

A.2.3.1 **Data Case Description:**

A.2.3.2 **CSF Audit:**

A.2.3.3 **Technical Review:**

A.2.3.4 **Contract-Problem/Non-Compliance:**

HWSS Reviewer: _____ Date:_____
Signature

Contractor Reviewer: _____ Date:_____
Signature

Verified by: _____ Date:_____
Signature

CASE #

SDG #

Contract Laboratory Program
REGION II/LABORATORY COMMUNICATION SYSTEM

Telephone Record Log

Date of Call: _____

ESAT Reviewer/Date: _____

Type of Analysis: ___Inorganic_____

Laboratory Name: _____

Lab Contact: _____

Call Initiated By: ___Laboratory _X_Region II

Inquiry made in reference to data for the following sample number(s):

Summary of Questions/Issues Discussed:

Summary of Resolution:

_____
Signature **Date:** _____

FIELD DUPLICATES

Sample No. **Field Duplicate No.** **Sample Matrix:**

Lab Code: **Case No. :** **SDG No.:**

% Solids Sample: **% Solids Duplicate:**

Concentration Units (ug/l or mg/kg dry weight):

	Action Limit	Sample Concentration	C	Duplicate Concentration	C	RPD	Difference	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Total/Dissolved Concentrations

Lab Code **Case No.** **SDG No.** **Sample Matrix: Water**

Concentration: ug/L

ANALYTE	TOTAL	C	DISSOLVED	C	DIFFERENCE	Q	M
ALUMINUM							
ARSENIC							
BARIUM							
BERYLLIUM							
CADMIUM							
CALCIUM							
CHROMIUM							
COBALT							
COPPER							
IRON							
LEAD							
MAGNESIUM							
MAGNESE							
MERCURY							
NICKEL							
POTASSIUM							
SELENIUM							
SILVER							
SODIUM							
THALLIUM							
VANADIUM							
ZINC							
CYANIDE							

CONTRACT LABORATORY PROGRAM
CLP RAS RE-ANALYSIS REQUEST/APPROVAL RECORD

SECTION A (TO BE COMPLETED BY REGIONAL SENDING OFFICIAL)

Initiated By:

Name, Affiliation, Phone Number

_____

Case Number: _____
- OLM
- OLC
- ILM

Details of Re-Analysis Request:

- Laboratory Name /Contract Number: _____
- Affected Sample Number(s) and Fraction(s): _____
- Reason for Re-Analysis: _____
- Contract Statement of Work Citation*: _____

_____

- Comments: _____

_____

- * PROVIDE SOW CITATION THAT SUPPORTS THIS REQUEST

RE-ANALYSIS Billable ( ) Not Billable ( )

- Approved By: _____ Date: _____

Authorized Regional Sending CLP PO Signature

SECTION B (TO BE COMPLETED BY SMO)

Name of SMO Contact _____ Date: _____

Date of Laboratory Notification (Verbal): _____

Re-analysis Start Date: _____ Data Due Date: _____

Return completed form to:
Sample Management Office (SMO)

Distribtion: (1) CLP PO Copy (2) Regional Sending Official Copy (3) SMO File Copy (4) Laboratory Copy
Final 9/3/99

CLP DATA ASSESSMENT SUMMARY FORM (INORGANICS)

Type of Review: _____ Date:_____ Case# _____SDG#___

Site: _____ Lab Name: _____

Reviewer's Initials:_____ Number of Samples:_____

Analytes Rejected (R) Due to Exceeding Review Criteria

	Holding Time	CRQL Std	Blanks	ICS	Spike Recovery	Dup. Lab.	Dup. Field	LCS	ICP Serial Dilution	% Solids	Internal Std. ICP-MS	Tuning ICP-MS	Total Analytes	Rejection %
ICP-AES														
ICP-MS														
Mercury														
Cyanide														
Total														

Analytes Flagged (J) as Estimated Due to Exceeding Review Criteria

	Holding Time	CRQL Std	Blanks	ICS	Spike Recovery	Dup. Lab.	Dup. Field	LCS	ICP Serial Dilution	% Solids	Internal Std. ICP-MS	Tuning ICP-MS	Total Analytes	Rejection %
ICP-AES														
ICP-MS														
Mercury														
Cyanide														
Total														